

The Journal *of the* SOCIETY OF DYERS AND COLOURISTS

Volume 71 Number 3

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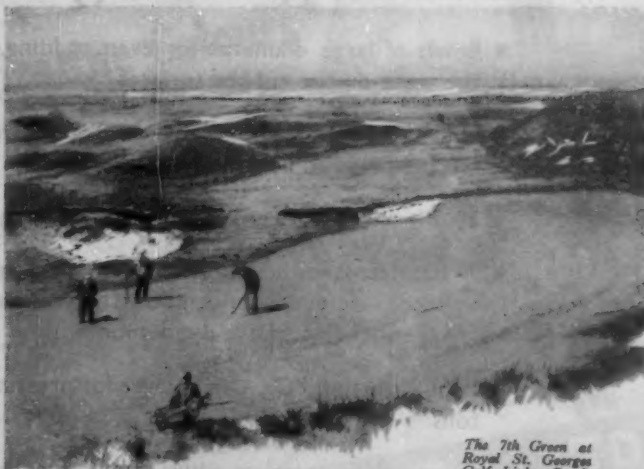
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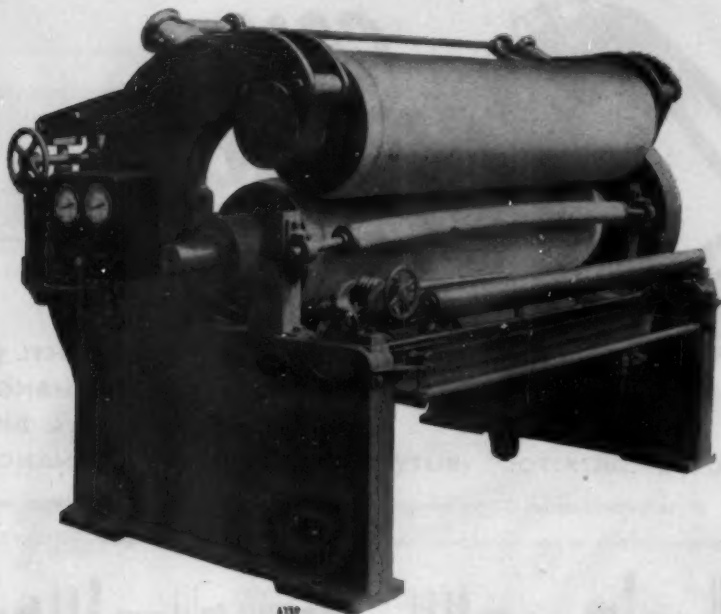
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NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1-6 of the January 1955 and pages 262-266 of the July 1954 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (Telephone Bradford 25138-9). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

COMMUNICATIONS

The Influence of the Physical State of Dyes upon their Light Fastness

G. Baxter, C. H. Giles, Miss M. N. McKee, and N. Macaulay

The Polarography of Azo Dyes

J. de O. Cabral and H. A. Turner

The Effect of Chlorine on Dyed Nylon

A. N. Davidson and R. Preston

The Preparation and Properties of Regenerated Cellulose containing Vinyl Polymers

II—Staining, Swelling, and Stiffness Characteristics

G. Landells and C. S. Whewell

Some Aspects of the Mechanical Finishing of Cotton Textiles

G. M. Nabar and G. P. Tawde

The Effect on Wool of Boiling in Aqueous Solutions

II—Solutions of Ammonium Salts with and without Ammonia

R. V. Peryman

EXPLANATORY PAPERS ON MODERN THEORY

An Introduction to the Theory of Wool Dyeing

I—Equilibrium Theory of Affinity

L. Peters

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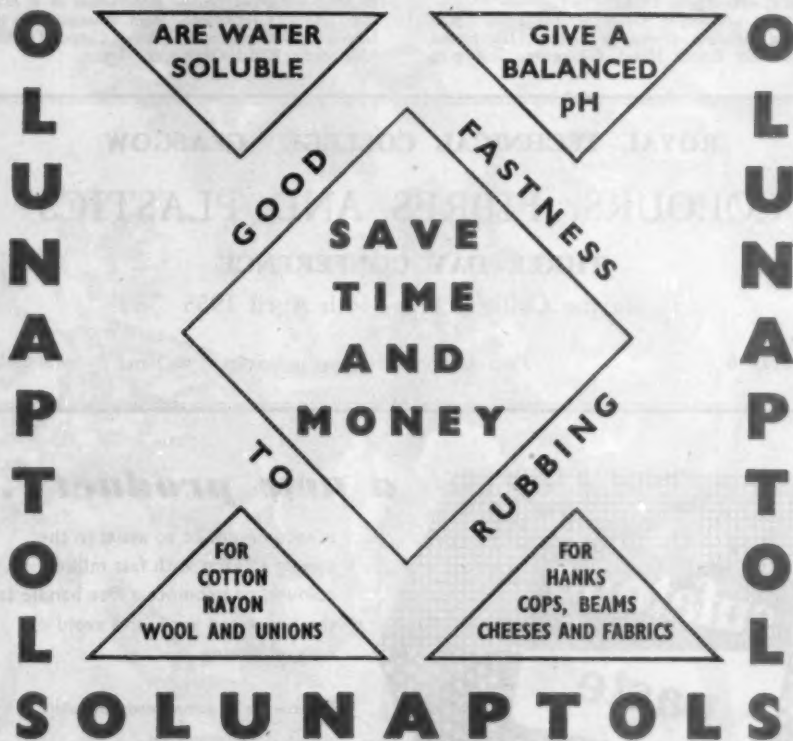
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FORTHCOMING MEETINGS OF THE SOCIETY

Thursday, 24th March 1955

WEST RIDING SECTION—ANNUAL GENERAL MEETING. The Victoria Hotel, Bridge Street, Bradford. 7.30 p.m.

Monday, 28th March 1955

LONDON SECTION—ANNUAL GENERAL MEETING 6-30 p.m. Followed by DINNER at 7-45 p.m. Aldwych Braserie, 1 Aldwych, London WC2.

Friday, 1st April 1955

ANNUAL GENERAL MEETING AND DINNER of the Society. Queens Hotel, Leeds.

Tuesday, 5th April 1955

SCOTTISH SECTION—ANNUAL GENERAL MEETING 7 p.m. *Primitive and Modern Dyeing Practice*. (Illustrated) (I.C.I. Ltd.). St. Enoch Hotel, Glasgow. 7.30 p.m.

Wednesday, 6th April 1955

MIDLANDS SECTION. *Some Recent Research in a Dyeing Laboratory*. Paper by G. H. Lister, Esq., B.Sc., Ph.D. (ANNUAL GENERAL MEETING at 6.30 p.m.). Kings Head Hotel, Loughborough.

Friday, 15th April 1955

MANCHESTER SECTION—ANNUAL GENERAL MEETING. "Courpleta" *The Dyeing and Other Properties of Triacetate Yarn and Staple*. J. Boulton, Esq., M.Sc. Tech., F.S.D.C., F.T.I. Textile Institute, Manchester. 7 p.m.

Thursday, 21st April 1955

MIDLANDS SECTION. *Some Experiences of a knitted-goods dyer*. A. W. Carpenter. (Joint Meeting with the Textile Institute Kidderminster Section). Carpet Trades Canteen. Mill Street, Kidderminster. 7 p.m.

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THE JOURNAL

OF THE

Society of Dyers and Colourists

Volume 71 Number 3

MARCH 1955

Issued Monthly

Proceedings of the Society

Metallic Pigments

G. W. WENDON

*Meeting of the West Riding Section held at the Hotel Metropole, Leeds, on 14th January 1955,
Mr. H. Foster in the chair*

The manufacture of metallic flake pigments is outlined. Their salient features are described and related to the more common applications. The production and the properties of dyed metal powders are discussed and contrasted with those of undyed metallic pigments as well as of ordinary pigments. Further examples are given of the combining of metallic appearance with spectral colours.

Metallic pigments are familiar from various kinds of application—lampposts, gasholders, and petrol storage tanks coated with aluminium paint, gas fires painted in gold colour, hatbands embossed with gold lettering, and many other examples.

The manufacture of these metallic pigments derives from the goldbeaters' art. This was purely manual work. Gold was progressively beaten or hammered until the final gold powder resulted. Bronze powder was probably the first metallic pigment mechanically produced on a sizable scale. It was overtaken in output and importance by aluminium powder during the early parts of this century. The large-scale manufacture of these powders was carried on mainly in Germany until that time. Bessemer had attempted to produce aluminium powder in this country in the nineteenth century, but the process was discontinued after a relatively short time. The picture is very different today. Although Germany is one of the more important producing countries, Britain has gained ascendancy in Europe in regard to aluminium paste, the preferred pigment for aluminium paint. Apart from Britain and Germany, the United States and Canada are the principal producing countries, American output exceeding that of any other country. Moreover, there are factories in Switzerland, France, Belgium, Holland, Sweden, Italy, and Austria.

There is an old proverb that "All that glitters is not gold". It certainly applies to the subject of this paper. In the metallic pigments industry and trade, cases of mistaken identity arise very frequently from loose and inaccurate nomenclature. In other words, all that is called gold and silver powder is not gold and silver. Real gold powder is

used for high-class decorative paints and other coating compositions. Its field of application is restricted by its high cost. Real silver powder is made on a small scale. It is hardly ever used for pigmenting paints and lacquers, but rather for colouring watch dials and the like. What are popularly known as "silver" powder and paint are, in fact, aluminium powder and aluminium paint.

All these metallic pigments were formerly referred to generically as "bronze pigments"; and even today they are often described like this, i.e. as "gold bronze powder", "copper bronze powder", "aluminium bronze powder". The uninitiated may be forgiven if he concludes that these pigments are powdered bronzes. They are, however, nothing of the kind. What they have in common is the lamelliform, flake-like structure of their particles. So-called "gold bronze powder" or, simply, "bronze powder" is brass flake powder; so-called "copper bronze powder" and "aluminium bronze powder" are nowadays described more accurately as *copper flake* and *aluminium flake* powders. These two are the products of the comminution of elementary metals, not of alloys.

MANUFACTURE

Bronze powder is made either from splashings, obtained from the molten metal, or from atomised brass powder. When splashings are used, they are usually flattened in stamping pots to an intermediate product known as *flitter*. This is already a lamellar material, and can vary from coarse flitter, perhaps 6 or 8 mesh, to fine grades, about 40 mesh. The flitter is hammered in smaller stamps or in ball mills to the final bronze powder. Atomised or

spherical brass powder is hammered, as a rule, in ball mills in a single operation.

Since the large-scale development of industrial metallic paints, the output of aluminium pigments has exceeded by many times the total output of all the other metallic flake pigments put together. Aluminium flake powder is either made from splashings, as is bronze powder, or from aluminium foil scrap, which is first shredded. Again, aluminium flake powder can be made in ball mills or stamps, more frequently in ball mills nowadays.

For the past 15–20 years, aluminium flake pigments have been available also in paste form, and are preferably used in this form in the manufacture of paints, lacquers, and printing inks. This aluminium paste is always produced in ball mills by a wet-milling process, from either shredded aluminium foil or atomised powder. The raw material is hammered in a liquid medium, generally white spirit, the resulting sludge filtered, and the filter-cake adjusted in consistency by the addition of fresh liquid.

In all these processes for the comminution of brass, copper, and aluminium, a lubricant has to be added to prevent welding of particles. The lubricant most widely used is commercially pure stearic acid.

When aluminium is hammered dry in ball mills, these are filled with a controlled inert atmosphere, consisting largely of nitrogen and carbon dioxide, with a small percentage of oxygen, to overcome the danger of self-ignition. The use of controlled atmospheres is evidently impracticable in the open stamping pots. When the metal is reduced by wet-milling in white spirit, the provision of inert gas is unnecessary, the liquid giving the required protection.

Before they can be used for bright paints, aluminium and bronze powders have to undergo a polishing operation. This is normally carried out in cylindrical drums fitted with radially arranged brushes. These are pressed against the internal surface of the drum by means of springs, and they polish the lamellar particles. An additional amount of stearic acid is incorporated at this stage. Stearic acid is used, as a rule, because it is solid at ordinary temperatures, but melts at the temperatures prevailing inside the milling and polishing drums, and also because of the tendency towards orientation characteristic of fatty acids.

Aluminium paste pigments are usually manufactured without any polishing operation. However, they can have excellent colour and metallic lustre. It would seem that the conditions under which surface forces come into play are rather different in dry and wet systems.

PROPERTIES

Metallic flake powder (and paste) is the odd man out among the pigments, and this is probably due more to the lamellar structure of its particles than to the chemical properties of the metals concerned. The chemical properties of these metals certainly become evident at many stages from manufacture

to application, but a consideration of their physical and physicochemical properties will show immediately the peculiar features of these products.

Aluminium flake powder, for instance, differs sharply from granular or atomised aluminium powder in many of its physical properties, and it is due to these special characteristics that aluminium flake powder is the only type of aluminium powder suitable for the pigmentation of paints, printing inks, and other coating compositions.

The true density (specific gravity) of aluminium is 2.70. If allowance is made for the content of lubricant and surface oxide, it will be found that the average value for the density of aluminium flake powder is about 2.55. The bulk (or apparent) density of the lamellar powder is only a fraction of this value, owing to the large volume of air held by the powder. In this respect, flake powder differs sharply from atomised powder. While aluminium flake powder has apparent densities of 0.3–0.7 in the dry state, the corresponding figures for atomised powder are approx. 1.0–1.5.

If the diameter of the particles is considered, metallic flake powders are, on average, coarser than other pigments. The coarser grades of aluminium and bronze powder are about 100 mesh. The finest grades of metallic powder and paste pass almost completely through a British 300 mesh sieve or an American 325 mesh screen. However, the thickness of these particles is always less than one-fiftieth of the diameter, often one-hundredth or even less. The finest grades of aluminium paste pigments have a mean flake thickness of approx. 0.2μ .

COVERING POWER

The thickness of metallic flake powder is best calculated from another value, viz. the covering power of the powder. This is an entirely different kind of covering power from that given for paints. By the "covering power" of a paint is meant the area a given amount, usually one gallon, of it will cover in practice. This value cannot be stated very precisely, as it largely depends on the method of application and the technique of the operative. The covering power of a metallic powder always refers to the area of water which unit weight of that powder will cover. Owing to their lamellar structure and the water-repellent film of fatty acid carried by these powders, they float completely on water. It is therefore feasible to determine their covering capacity on water. Edwards and Mason¹ have developed a method in which a shallow, rectangular metal trough is filled with water and a weighed quantity of the powder is sprinkled on the surface. The powder is then spread out completely, and is carefully worked until a continuous film, one flake thick, is formed. This film is alternately compressed and allowed to expand. When it is compressed, wrinkles are formed, and these disappear on expansion. The end-point is the position in which these wrinkles just disappear.

The covering power of a metallic powder is therefore very different from the covering power of paint, although it remains true that a metallic

pigment with a higher covering capacity will yield a paint with a greater covering power, other things being equal, e.g. paint medium and technique of application.

The thickness of the metal powder can be calculated from the observed value for covering power and the known value for specific gravity. If t is the mean flake thickness in microns, A the area in sq. cm. covered by 1 g. of the powder, and d the true density, then—

$$t = \frac{10,000}{A \times d}$$

LEAFING POWER

The most interesting and perhaps most startling property of metallic flake powders is their so-called "leafing power". When e.g. aluminium powder or paste is mixed with a typical paint vehicle, e.g. oil varnish or bituminous medium, the small particles orient themselves at the air-liquid interface to form a continuous metallic film of mirror-like appearance. The lamellar particles arrange themselves in parallel layers surrounded by films of paint medium. This "leafing" phenomenon seems surprising, at first sight, in view of the fact that the metal powder has a much higher specific gravity than the paint vehicle. It is due to the combined effect of the flaky structure of the powder particles and the layer of fatty acid with which they are coated. This film of fatty acid is itself oriented, and this orientation is a condition of the orientation of the metallic particles in the paint medium, known as *leafing*. The fatty acid has therefore a dual function: it acts as a lubricant during milling, and subsequently as a polishing and a leafing agent.

The leafing ability of metallic pigments is of fundamental importance for their use in paints for surface coatings and in printing inks. Brightness and metallic lustre depend entirely on it. Non-leafing metal powders yield dull and lifeless paint films. In the case of protective aluminium paint for outdoor use, leafing power and bright finish are of greatest importance. Here, the function of the metallic paint is to reflect sunlight. The aluminium pigment protects the paint vehicle from the destructive action of the ultraviolet rays. Also, e.g. when applied to the outer surface of petrol storage tanks, it keeps their inside relatively cool, by reflecting sunlight, and reduces loss by evaporation. Practically all petrol storage tanks, at least in countries with hot climates, are therefore surfaced with aluminium paint nowadays.

For the development of leafing power, the powder must be composed of lamellar particles coated with a suitable polishing lubricant, normally a fatty acid. The milling technique must be so adapted as to yield flat and thin particles, for which it is essential that comminution of the metal is caused by impact and not by abrasion.

Secondly, the powder must have reached a sufficiently fine state of division. When an aluminium powder is separated into different size fractions by screening, and the leafing power of these fractions determined, it is found that the coarser particles show little leafing power, while the

finest fraction shows the most pronounced leafing tendency. On the other hand, if comminution is carried too far, the particles will tend to lose leafing power and, especially, metallic lustre.

A very important and interesting feature is the gradual development of leafing power in metallic pigments. When an aluminium or bronze powder is removed from the polishing drum, it is usually not adequate for use in paint or lacquer. It does leaf, but not to the extent required in its application. It is normal practice to let the powder mature for several months, during which its leafing power is much enhanced.

It had been assumed for some time, as a working hypothesis, that the chemical explanation of this ageing process lies in the slow interaction between the metallic or metallic oxide surface and the fatty acid. About six years ago, Trillat² provided evidence of the formation of an aluminium stearate in the case of aluminium flake powder. He submitted to X-ray analysis both freshly manufactured and aged aluminium powders. Also, he extracted one of the powders with ether, compared the X-ray patterns of the extracted and the unextracted powders, and examined the composition of the extract. Trillat found that the aluminium particle consists of an inner core of metallic aluminium surrounded by a zone of aluminium oxide-stearate; the outer layer consists of free stearic acid, and only this layer is oriented. The free stearic acid in the outer shell has a tendency towards crystallisation. The longer the powder is polished, the less this crystallising tendency is in evidence. It seems that the crystals are broken up during polishing.

The maturing period for aluminium paste is much shorter than for aluminium powder, nor is polishing required to promote leafing. Presumably, the liquid portion of the paste acts as a distributing medium for the fatty acid.

A problem peculiar to metallic paste pigments is stabilisation of leafing power. Metallic flake powders retain their leafing power indefinitely if stored under proper conditions, i.e. provided that extremes of temperature are avoided and moisture is excluded. Metallic paste pigments tend to lose leafing power on prolonged storage, sometimes after a few months, sometimes after a year, presumably owing to the presence of impurities in the liquid constituent of the paste that affect the aluminium flakes. It is therefore desirable to counteract these influences and stabilise the leafing power of the pigment. Aluminium pastes have been produced which are completely stable.

One reason for excluding moisture from drums containing aluminium powder or paste is to prevent chemical reaction leading to formation of hydrogen, either in the drum or in the paint into which the metallic pigment has been incorporated. Apart from that, prolonged exposure of aluminium powder to high water vapour concentrations is harmful to leafing and reflectivity. Experiments have shown that exposure to a saturated atmosphere for six months results in a considerably smaller increase in leafing power compared to the normal develop-

ment of the power. At the same time, it has been shown that storage in an intensively dried atmosphere has a similarly bad effect on leafing. It would seem that a very small amount of water vapour, such as is always present in ordinary air, is essential to assist the development of leafing power, perhaps by some catalytic action.

Leafing power can be measured qualitatively by mixing the metallic flake pigment with a suitable paint vehicle and noting the formation and appearance of the metallic mirror. The paint should be applied to metal plate, and the appearance of the dried paint film judged against some standard.

This type of test has its obvious shortcomings. In the first place, it is not quantitative. Secondly, it depends on the nature of the paint medium used and the skill of the operative. Edwards³ therefore developed a quantitative method for measuring leafing power. In this, a steel spatula of standardised dimensions is immersed in a mixture made up of the aluminium pigment and a solution of coumarone resin in white spirit. It is slowly withdrawn, and allowed to dry in a draught-free atmosphere. The lower part of the paint film formed on the spatula will be continuous. Near the top, however, cracks will be visible. The percentage leafing power is 100 times the ratio of the depth of the area free from cracks to the total depth of immersion.

The advantage of this test is that it is quantitative and standardised. Its limitations are first the somewhat artificial nature of the medium and, secondly, the lack of facility for judging colour and reflectivity. The British Standard for aluminium pigments⁴ incorporates both the Edwards leafing test and a qualitative test as outlined before.

The measurement of the reflectivity of metallic paint films has been attempted by the use of a variety of reflectometers. So far, equipment has not been standardised for this purpose. In the last resort, the observer's eye must be the final judge of the colour and the gloss of metallic paint films.

It should be stressed that the best metallic pigments will not exhibit good leafing if the paint vehicle is not suitably formulated. Owing to their difference from other pigments, it is necessary to adjust the paint vehicle accordingly. A paint medium may be excellent for making green, yellow, blue, or brown paint and quite useless for producing aluminium or bronze paint. The converse is sometimes true. Vehicles for metallic paint should have low acid number and contain only a moderate amount of drying oil. The viscosity should be kept fairly low. Solvents should be chosen for high surface tension from among the chemically inert hydrocarbons. A proportion of aromatic solvents aids leafing. Cobalt driers only should be used: most others are more or less harmful to the preservation of leafing power.

High leafing power is essential when aluminium paint is intended for reflecting sunlight. On the other hand, leafing is unnecessary when the aluminium pigment is used for primers. In fact,

non-leafing grades are often chosen to prevent the metallic paint from shining through the finishing coat, especially when the latter is of light colour. Between these extremes of high reflectivity and dull grey finish lies an intermediate range. Cellulose lacquers are usually pigmented with leafing grades of metal powder or paste, but these vehicles do not promote leafing very strongly and yield a rather more "frosted" finish. Metallic powders used in lithographic work are frequently non-leafing. Leafing power is not essential for brightness in this case, especially if the powder is dusted on a sized surface.

COLOUR

The colour of bronze powders, i.e. brass flake powders, depends on the chemical composition of the raw material used in their production, notably the proportion of copper to zinc. The yellowest brass that can be hammered satisfactorily contains about 70% copper and 30% zinc.

Aluminium flake powder exhibits the slightly bluish cast characteristic of aluminium. This bluish cast is probably more obvious in the powdered state. Occasionally, demands are made for an aluminium powder with a so-called "silvery" appearance. It is possible to neutralise the bluish cast and produce a pigment with a colour more reminiscent of silver by mixing aluminium powder with gold bronze powder. A range of powders of colours from light to antique silver can be made in this way.

The lustre of the metallic paint film is generally related to the leafing ability of the pigment. However, high leafing power by itself is not necessarily a criterion of good colour and reflectivity. Leafing power is only one of several factors in a complex system that is as yet imperfectly understood. All that can be stated is that, other things being equal, high leafing power will give rise to the formation of a brighter and "whiter" paint film. Instances are, however, met in practice of the metallic pigment with the lower leafing power yielding the brighter paint film.

Aluminium pigments intended for incorporation into the typical bright metallic paint should be highly leafing, reflective, and as light in colour as possible. In many fields of application, however, e.g. for most plastics, the main requirements are brightness and light colour, leafing being often unimportant. In other cases, again, dark shades of aluminium powder are in demand, as for cosmetic creams for eye-shadows.

The most striking way of combining metallic appearance with the colours of the spectrum is undoubtedly shown by dyed metal powders, of which dyed aluminium powder is the best known example. This type of powder is extremely brilliant and combines bright colour with metallic lustre. Its function is purely decorative. It is mainly used for fancy work.

The dyeing of metal powders presents peculiar difficulties, and the dyeing processes differ in many ways from more common dyeing methods.

The dyeing of aluminium in solid or block form was developed and perfected during the late thirties and early forties. This is an application of the anodising process. The aluminium is made the anode of an electrolytic cell, and during, or immediately following, its anodic oxidation it takes up dyes present in the electrolytic bath.

This process is not applicable to the dyeing of aluminium powder, because the powder is a non-conductor of electricity at normal voltages, each flake carrying layers of fatty acid and metallic soap. It is possible to remove the outer shell of free fatty acid, but it is not feasible to break down the intermediate oxide-stearate layer so as to make the powder conducting at the voltages in question. Also, there would be mechanical difficulties arising from the structure of the metallic pigment if it was attempted to make it the electrode of an electrolytic cell system.

In practice, aluminium and other metallic flake powders are dyed by processes consisting of several stages. They cannot be dyed directly by treatment with dye solutions, owing to the presence of the barrier film of fatty acid. This has to be removed, and a mordant film established on the metallic particles that renders them capable of absorbing dyes. There is a fairly wide range of mordants available for this purpose. When the powder has been dyed, it is dried slowly, and can then be polished, if necessary, in the conventional type of polishing drum used in the metal powder industry.

It is regrettable that it has not proved practicable so far to apply the fastest types of dye to these powders without the loss of metallic lustre, although a certain progress has been made in this field. Even among the classes of dyes that can be fixed to metal powders, the choice depends ultimately on the kind of limitation acceptable to the user of the dyed powder. Unfortunately, the brighter dyes are rarely very fast to light, and the fastest are not usually very bright. There would be little point, however, in applying a light-fast dye to metallic flake powder if it produced a dull appearance devoid of the gloss and sparkle normally associated with this material.

The other limitation noticeable in dyed metal powders concerns their limited compatibility with paint vehicles and their surface properties in general. As a result of the preliminary treatment given to the powder before dyeing, which results in the displacement of the fatty acid film, the leafing power is destroyed. Even if the dyed powder is subsequently polished with stearic acid, the leafing power is not restored. Polishing can only enhance the gloss of the powder and increase its resistance to the action of solvents and atmospheric influences. In many cases, polishing is of no advantage and is therefore dispensed with. This is particularly true when the powder is to be incorporated into plastics.

Since dyed metal powders do not leaf, i.e. since the fatty acid applied to them does not form the kind of oriented film required for leafing, the conclusion seems compelling that the interaction

between the metallic part of the particle and the fatty acid layer is a necessary condition for the development of leafing. In that case, the film of dye separating the metallic core from the fatty acid layer would prevent this surface reaction and the generation of leafing power.

Because of these two shortcomings, viz. absence of leafing power and limited resistance to light, coloured metal powders should be used only for interior application. As they do not leaf, they cannot be incorporated into the typical oil or bituminous varnish, the strong and dark colours of which they are unable to hide. In any event, dyed metal powders are not easily dispersed in these types of vehicle. The media mainly used are cellulose lacquers, shellac, and some other types in which leafing is unimportant.

It may seem strange that aluminium powder is frequently dyed in gold and copper colours, seeing that there are natural metal powders available in these colours. The reason is that there are some applications for which copper and its alloys are unsuitable. Copper and brass flake powders are more susceptible to chemical attack than is aluminium powder, and they tend to turn green. Because of their reaction with sulphur, they cannot be incorporated into vulcanised rubber without discoloration. There are a number of other cases in which the tendency of copper and bronze powders to be oxidised is objectionable. Aluminium powder dyed in copper or bronze colours does not suffer from this disadvantage.

Secondly, the effect of copper and bronze powders on most cellulose-type lacquers, causing gelling often within a few hours, has frequently led to their replacement by dyed aluminium powder.

Thirdly, the rather high density of copper and bronze powders severely restricts their use in pigmenting plastics, owing to their high rate of settling. They remain dispersed in the compositions insufficiently long to impart the desired metallic appearance.

Lastly, it is sometimes an important point that the amount of dyed aluminium powder needed for pigmentation is only a fraction of the amount of bronze powder, again owing to the difference in density.

For exterior application, the polychromatic type of paint is a much more satisfactory if less striking example of the combination of metallic appearance with the spectral colours. These paints differ from ordinary paints in containing small amounts of natural aluminium flake powder or paste that impart metallic sheen to the finish. The metallic effect thus obtained is more discreet than that obtainable from dyed metal powder. It varies from a slight metallic sparkle to a soft silky sheen, according to the grade of aluminium pigment used.

The concentration of aluminium powder or paste in polychromatic paint is rather low, normally 1-3% by weight of the paint. This compares with concentrations of the order of 15-20% for straight aluminium paint. The aluminium pigment used in

polychromatic paint and lacquer should not have much leafing power. Unless any leafing capacity that it may have is destroyed during the production of the paint, there is a risk that the bright aluminium flakes orient themselves at the surface and form continuous metallic areas instead of remaining dispersed as discrete particles.

The aluminium pigment is never ground into the paint. Dispersion is obtained simply by stirring. Grinding metallic flake powder would lead to loss of metallic lustre, and is therefore always to be avoided in the manufacture of any kind of metallic paint intended for surface coatings.

As it is possible to combine metallic appearance with the colour of light-fast pigments, polychromatic paints lend themselves remarkably well to outdoor use. One of their most important applications is the surfacing of motorcar bodies.

Another way of producing a coloured metallic appearance consists in applying a dyed transparent lacquer over a bright natural aluminium paint film. The effect obtained in this way is

striking. However, dyes that can be used for this kind of finish are not nearly as fast to light as the pigments found in polychromatic paint.

The systematic study of metallic flake powders goes back only a few decades. This is a very short time compared with that devoted to the study of the more conventional pigments and dyes, and will explain the relative ignorance that still exists in the field of metallic pigments.

ENGLISH METAL POWDER CO., LTD.

WEST DRAYTON

MIDDLESEX

(MS. received 27th October 1954)

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COMMUNICATIONS

The Affinities of Vat Dyes in Relation to their Constitutions

R. H. PETERS and H. H. SUMNER

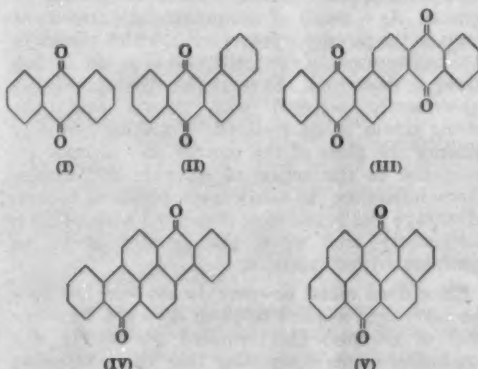
The affinities of a series of anthraquinone and carbocyclic vat dyes have been determined. The attraction for the fibre appears to arise from two main causes—polar or hydrogen bonding and non-polar or van der Waals forces. The results are analysed in terms of these. It is found that, in a series of related compounds, a relationship exists between the affinity and the van der Waals forces as determined by the extinction coefficients of the dyes.

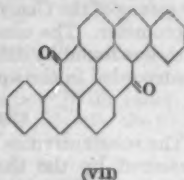
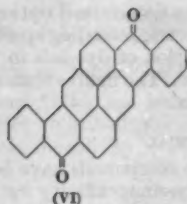
INTRODUCTION

Although the dyeing of cellulosic fibres with vat dyes is of great importance in practice, little is known about the relationship between the substantivity of such a dye and its constitution. This arises partly from the lack of available quantitative data, and partly from the difficulties involved in such a study. This is to some extent true also of the direct cotton dyes, as a result of the arbitrary conditions under which earlier experiments were carried out, dyes merely being classified as either non-substantive or substantive. Such a classification does not bring out the finer differences between the adsorption properties of various compounds.

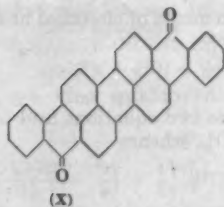
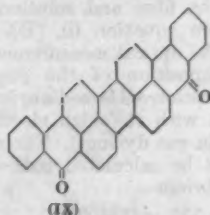
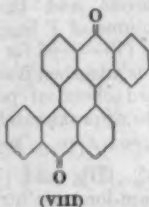
However, in spite of this, the properties required to confer substantivity on a direct dye are fairly well established¹. Briefly these are that the dye must—(a) be linear in shape, (b) be capable of assuming a planar configuration, and (c) contain groups capable of forming hydrogen bonds with the hydroxyl groups of the cellulose. It has been shown that the affinity is improved if—(a) the hydrogen-bond-forming groups are spaced at 10.3 Å., corresponding to the repeat spacing of the cellobiose units in cellulose; (b) the dye molecule contains a system of conjugated double bonds, which by resonance promotes coplanarity of the

molecule, and which also probably favours formation of hydrogen bonds by the terminal groups of the conjugated chain; (c) there are present the minimum number of solubilising groups necessary for solubility; and (d) the solubilising groups are disposed along one side of the dye molecule and the hydrogen-bonding groups along the other. The overall picture of the dyeing process is therefore of a long planar dye molecule adlineating itself compactly along the cellulose chains and being attached thereto by means of hydrogen bonds.



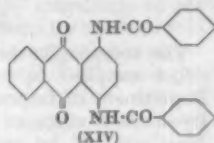
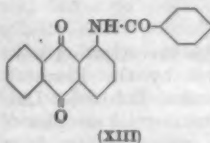
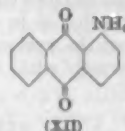
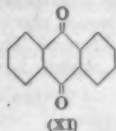


Unfortunately, when vat dyes are considered, such a mechanism cannot be invoked. The molecules in general are not linear in shape, and not all of them contain hydrogen-bond-forming groups. Their substantivity has been associated by Thompson² with their molecular complexity; e.g. in the series of compounds I-V, the first three show little affinity for cellulose, whilst IV and V do. Further, the position of the keto groups in the quinone is of importance, since the isomers VI and VII show a difference, VI being substantive whilst VII is not. Again, a substance (VIII) with fewer condensed nuclei is non-substantive. Finally, it may be noted that the long molecules IX and X both show high substantivity.

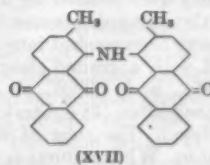
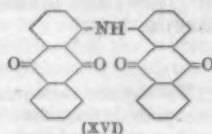
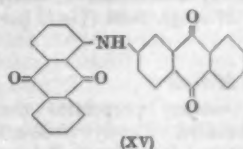


Apart from such systems of fused benzene nuclei, a large group of the compounds used as vat dyes are anthraquinone derivatives. A satisfactory explanation of the substantivity of these compounds is possible, since the substituents contain polar groups capable of forming hydrogen bonds with the fibre, as a comparison of the relative substantivities of the following compounds would suggest. In the series XI-XIV the first two have no substantivity, whilst the last two, which contain amido groups, do possess substantivity.

A similar effect is shown by azoic coupling components, where the amido group appears to be essential.



However, apparent anomalies do occur. Lukin³ states that XV and XVI have an affinity for



cellulose, whilst XVII has none, a difference which can be explained by the non-planarity of XVII arising from steric interaction between the methyl groups.

It is clear from the few compounds quoted here that hydrogen bonding by the substituent groups in the molecule cannot be the only source of substantivity, and other types of bonding must also be postulated. It has been suggested, for example, that the phenolic groups present in the leuco form of a vat dye may account for substantivity, but this is unlikely, since simple anthraquinone derivatives would show a small but definite affinity. This is not the case, however. Even when groups are present in the compound which are capable of being attached to the fibre by means of polar or hydrogen-bonding forces, as with substituted anthraquinones, it seems unlikely that there are sufficient present in many of the molecules (e.g. XVI or XIII) to endow them with a high attraction for the fibre. Modes of attachment in addition to polar forces must therefore be postulated, the most likely being the non-polar van der Waals type, which can arise when complex aromatic molecules are brought into contact with substrates. These forces are short-range in nature and additive over all the atoms in the molecule. They would account for the fact that substantivity increases with the complexity of the molecule, and is sensitive to its planarity or otherwise, since the attraction will be a maximum when maximum contact between substrate and adsorbed molecule

is achieved. The properties of this type of adsorption have been summarised by de Boer⁴.

With these ideas in mind it was decided to collect more quantitative data on the adsorption of vat dyes by cellulose in order to assess the contributions from the various kinds of forces. This paper is concerned with the data which have been collected.

EXPERIMENTAL

Materials

The compounds used in this investigation were either supplied in pure form by the Research Department of Imperial Chemical Industries Ltd. Dyestuffs Division or were commercial samples of dyes purified by repeated crystallisation from suitable solvents. The cellulose used was Bemberg (cuprammonium rayon) yarn (120 denier), which was scoured for 20 min. in a solution of soap (3 g./litre) and ammonia of sp.gr. 0.88 (1 ml. per litre) at 60°C.

Dyeing Method

To secure adequate vatting properties the compounds were first ball-milled. The dye (0.05 g.) and water (2 ml.) were rotated for approx. 2 days in a 1-oz. bottle containing some 10 glass balls. The contents were then washed into a conical flask (250 ml.) fitted with a ground-glass stopper, and reduced at 40°C. with 50 ml. of a solution of sodium hydrosulphite (48 g./litre) and caustic soda (19.56 g./litre). The time of vatting ranged from 15 to 60 min. When reduction was complete, water was added to make the volume up to 150 ml. After a lapse of 10 min. to enable this dyebath to reach the temperature of the thermostat (40°C.), the Bemberg yarn (0.25 g.) was added and the flask stoppered. When the dyeing had proceeded for 30 min., 10 ml. of sodium chloride solution (250 g. per litre) was added, and then the dyeing continued for a time sufficient to enable equilibrium to be reached (i.e. until no increase in the amount of dye adsorbed was observed on continued dyeing). The times required ranged from 1 to 8 hr. The preparation of the dyebath and that of the dyeings were both carried out under nitrogen to avoid air oxidation of the leuco dye solution.

After dyeing, the material was removed from the dyebath and squeezed to remove as much entrained liquid as possible. It was then allowed to oxidise in the air, washed with water, treated in 0.2% hydrochloric acid for 2 min., and finally rinsed in distilled water. After being pressed as dry as possible, the fibre was conditioned at 65% R.H. for at least 24 hr. before being weighed for the estimation of the dye. This was to ensure that the amount of material used was known accurately. Independent experiments showed that the error due to residual entrained dye liquor was of the order of 2%, and as such it was considered negligible.

The quantity of dye adsorbed was estimated by stripping the dye from a known weight of the dyed fibre at 40°C. with an aqueous solution of 50% Cellosolve containing sodium hydrosulphite (10 g./litre) and caustic soda (7.2 g./litre). The amount

of dye in the strippings was determined optically by means of the General Electric recording spectrophotometer. The concentration of dye left in the bath was found by difference. The figures obtained are recorded in the appendix.

THEORETICAL

The substantivities of the compounds have been measured by the thermodynamic affinity rather than by a substantivity ratio, which merely gives the ratio of the dye on the fibre to the total amount initially in the bath. The reasons for this choice are the arbitrary nature of such a ratio and the fact that the affinity is independent of varying dyebath conditions, depending only on the temperature of dyeing. The concentrations of dye in the bath and on the fibre are given in the appendix, and from these figures the values for the affinities have been computed by the method of Fowler, Michie, and Vickerstaff⁵, later modified by Peters and Simons⁶ to take into account the adsorption of the hydroxyl ions which are present in vat dyebaths. The method will be briefly outlined here.

The dye-fibre system is divided into two phases—the aqueous and the cellulosic—the latter having a volume of *V* litres per kilogram of fibre ascribed to it (*V* = 0.65 for Bemberg yarn⁷). When a dye Na₂D is adsorbed from the solution the change in standard chemical potential $\Delta\mu_D^\circ$ per mole of dye will be approximately—

$$-\Delta\mu_D^\circ \approx RT \ln [Na]_f^2 [D]_f - RT \ln [Na]_b^2 [D]_b \quad (i)$$

where $[Na]_f$, $[Na]_b$, $[D]_f$, and $[D]_b$ are the concentrations (in gram-ions per litre) of sodium and dye ions in the fibre and solution phases. For computing from equation (i), $[D]_f$ and $[D]_b$ are known from the optical measurements, and $[Na]_b$ from the composition of the original dyebath (since the adsorption of the sodium ions is negligible in comparison with the high electrolyte concentrations used in vat dyeings). The value of $[Na]_f$, however, must be calculated indirectly from the Donnan equilibrium—

$$[Na]_f [Cl]_f = [Na]_b [Cl]_b \quad (ii)$$

and the requirements of electrical neutrality in the cellulose phase—

$$[Na]_f = [Cl]_f + z[D]_f \quad (iii)$$

when the bath contains only dye and sodium chloride. These two equations lead to a quadratic equation in $[D]_f$, whence—

$$[Na]_f = [D]_f \left\{ \frac{z}{2} + \left(\frac{z^2}{4} + \frac{[Na]_b [Cl]_b}{[D]_b^2} \right)^{1/2} \right\} \quad (iv)$$

In order to apply this theory (in a simple manner) to vat dye solutions, Fowler *et al.*⁵ assumed that anions other than chloride in the vat dyebath could be replaced for the purpose of calculation by an equivalent quantity of chloride ions—

$$[Cl]_{b, equiv} = [Cl]_b + [OH]_b + 2[S_2O_4^{2-}]_b \quad (v)$$

where $[OH]_b$ and $[S_2O_4^{2-}]_b$ are the concentrations of hydroxyl and hydrosulphite ions in the bath. When, in addition, the adsorption of the hydroxyl ions is taken into account⁸, the overall effect is to modify equation (iv) to—

$$[Na]_f = [D]_f \left(\frac{z}{2} + \left(\frac{z^2}{4} + \frac{[Na]_s([Cl]_s + 2[S_2O_3]_s + K[OH]_s)}{[D]_f^2} \right)^{1/2} \right) \quad (vi)$$

where, as before⁶, $RT \ln K = -\Delta\mu_{NaOH}$.

Once $[Na]_f$ has been calculated, it is a simple matter to substitute in equation (i) in order to obtain the affinity, $-\Delta\mu_D$, of the dye for the fibre.

RESULTS AND DISCUSSION

The affinities of the dyes, which are given in the last column of the appendix, range from approximately zero to 6 kcal./mole. These values can be used to substantiate the suggestion of Thompson² that substantivity (here given by the numerical value of the affinity) depends on molecular complexity. As an approximate measure of the latter the molecular weight of the dye has been taken. The result of plotting affinity against molecular weight is given in Fig. 1, where some correlation can be seen between the two, the affinity increasing with the molecular weight. The agreement is obviously only rough, since related series of compounds with identical molecular weights have widely different affinities, as may be seen in Table I.

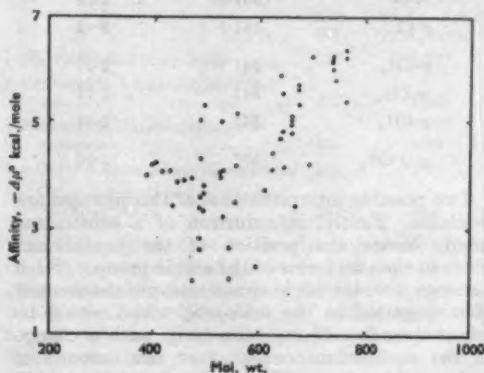


FIG. 1

TABLE I

Affinities of Substituted 1:4- and 1:5-Dibenzamidoanthraquinones
 $C_{22}H_{16}O_4(NH-CO-C_6H_4-R)_2$

Substituent R	Mol. Wt.	Affinity ($-\Delta\mu^\circ$, kcal./mole)	
		1:4	1:5
H	446	4.20	3.57
o-Cl	515	3.42	2.69
m-Cl	515	4.98	4.17
p-Cl	515	5.20	4.37
o-CH ₃	474	3.06	2.71
m-CH ₃	474	4.56	3.77
p-CH ₃	474	4.57	3.73
o-O-CH ₃	506	—	2.39
m-O-CH ₃	506	—	4.86
p-O-CH ₃	506	4.44	4.11

The affinity which has been measured is in fact the difference between those of the dye for the fibre and for the solvent, so that any change in the latter will be reflected in the measured quantity.

This aspect has been discussed at some length by Meggy⁸, who has suggested that the prime reason for the affinity of dyes for fibres lies in the fact that, when a dye is removed from the aqueous phase and put on to a substrate, there is a reduction in the area of interface between the hydrophobic part of the dye molecule and the water, which liberates sufficient energy to enable the dye to attach itself to the fibre. However, although this may be a very real factor in determining the magnitude of the affinity, and is possibly the reason for the increased affinity of, for example, 1-N-methylaminoanthraquinone as compared with 1-aminoanthraquinone, the major differences observed between dyes of closely similar structures suggests that some consideration must be given to the mode of attachment of dyes to the fibre. For example, the difference between 1-benzamidoanthraquinone and 1-benzylaminoanthraquinone is large (2.37 and < 1 kcal./mole respectively), and it seems unlikely that the leuco forms of these two compounds will have markedly different affinities for the water phase. In fact, if there is any difference, the latter compound would probably have the lower solubility and hence the greater affinity for the fibre, which is not so. Further, in a series of dyes of the type—

Anthraquinone-NH-CO-(CH₂)_n-CO-NH-Anthraquinone where n ranges from 2 to 8, the solubility will decrease as the hydrophobic nature of the compound increases, i.e. as n increases. However, no trend is noted in the affinities, which are all approximately the same (Table II). It must be assumed, therefore, that there is some more definite source of affinity of the dye for the fibre.

TABLE II

Affinities of NN-Di- α -anthraquinonylsuccinamide and its Homologues
 $C_{42}H_{30}O_8NH-CO-(CH_2)_n-CO-NH-C_{42}H_{30}O_8$ ($n = 2-8$)

n	Mol. Wt.	Affinity, $-\Delta\mu^\circ$ (kcal./mole)
2	528	3.59
3	542	3.63
4	556	3.64
5	570	3.46
6	584	3.46
7	598	3.28
8	612	3.53

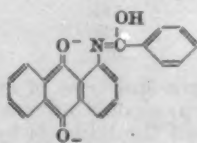
For this two principal types of forces must be considered, the non-polar or van der Waals bonding forces, and the dipolar or hydrogen bond type. If, as mentioned previously, the dye is attached to the fibre by means of forces of the van der Waals type, these will be larger the greater the conjugation in the molecule and the greater the area of contact with the fibre. Hence, provided that a planar dye molecule gives maximum contact, and that all the dyes in a particular series are equally planar, dyes of closely similar structure should have nearly equal affinities. This is not the case, as is indicated in Table I, and it is clear that polar (e.g. hydrogen bond type) attractions are also playing an important part. An example of the relative importance of these two kinds of bonding comes from calculations of the energy of adsorption

of phenol on crystals of sodium chloride⁴. For this system it was calculated that the dipolar or hydrogen bonds involving the OH group accounted for slightly less than 60%, and the non-polar forces for 40%, of the energy of interaction. It must therefore be assumed that leuco vat dyes, which contain several condensed aromatic nuclei, and in many cases one or more groups capable of forming hydrogen bonds, may be adsorbed by virtue of both non-polar and polar forces; and it is on this basis that the results obtained in this work are discussed, under the headings of (a) monosubstituted and (b) disubstituted anthraquinones, and (c) other compounds.

(a) Monosubstituted Anthraquinones

Anthraquinone and its simple derivatives, e.g. 1-chloro, 1-methyl, etc., are adsorbed to only small extents on cellulose, having affinities below 1 kcal./mole. The two groups necessary to produce an appreciable affinity are the anilino and the acylamino. Thus 1-(2:4-dimethylphenylamino)-anthraquinone has an affinity of 2, and the 1-acetamido, 2-acetamido, 1-benzamido, and 2-benzamido-anthraquinones have values of $-\Delta\mu^\circ$ of 1.52, 1.53, 2.37, and 2.05 kcal./mole respectively. When compared with anthraquinone, most of the increase in affinity of the acetamido derivatives must lie in their hydrogen-bond-forming capabilities, since there is only a comparatively small increase in the area of the molecule or in the length of the conjugated chain.

Replacement of the methyl by a phenyl group enhances the affinity (cf. 1-acetamido- and 1-benzamido-anthraquinones), presumably because the phenyl residue will not only affect the electron density at the amido group and hence its hydrogen-bond-forming ability, but will also supply a group possessing considerable van der Waals attraction for the fibre. It is also interesting to note here that if the amido group is in the iminol form—and unpublished evidence from a study of the colour of these leuco derivatives by Peters and Sumner supports this—then a conjugated chain can be set up which includes the whole molecule, e.g.—



Such an increase in the conjugation of the molecule, according to Schirm's hypothesis⁵ for direct cotton dyes, would lead to an increase in affinity. This may mean that increasing conjugation increases the number of π electrons or the polarisability of the molecule, and hence the non-polar bonding possibilities. There is no doubt that the benzamido group is a much more effective substituent than the acetamido. The importance of the amido $-\text{NH}-\text{CO}-$ group, and particularly the benzamido, in conferring substantivity has been observed previously for direct cotton dyes and azoic coupling components. Here, again, the possibilities of

iminolisation have been considered. In the case of the latter type of compound, 3-carboxy-2-naphthol and 3-amino-2-naphthol have no affinity, whilst the corresponding anilino and benzamido derivatives have affinities of 1.88 and 1.65 kcal./mole respectively.

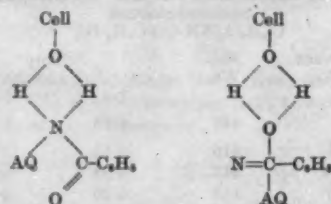
The affinity of 1-benzamidoanthraquinone may be modified by the introduction of substituents into the benzene ring of the side-chain. Thus groups such as chloro, methyl, and methoxy, although they do not have any specific attraction for the fibre, all result in a marked increase in the affinity (Table III).

TABLE III

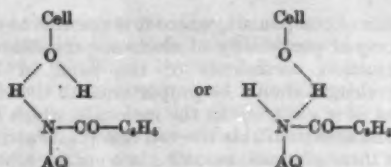
Affinities of Substituted
1-Benzamidoanthraquinones
 $\text{C}_{14}\text{H}_9\text{O}_2\text{NH}-\text{CO}-\text{C}_6\text{H}_4\text{R}$

Substituent R	Mol. Wt.	Affinity, $-\Delta\mu^\circ$ (kcal./mole)
H	327	2.37
o-Cl	361.5	1.98
m-Cl	361.5	2.73
p-Cl	361.5	3.02
o-CH ₃	341	2.07
m-CH ₃	341	2.74
p-CH ₃	341	2.68
p-O-CH ₃	357	2.90

Two possible interpretations of this increase are available. Firstly, introduction of a substituent could change the position of the equilibrium between the two forms of the amido group. Such a change would result in an alteration of the amount of conjugation in the molecule, which would be reflected in the affinity. Similarly, such a change in the equilibrium could affect the amount of hydrogen bonding. The bonding of the amido group to the hydroxyl groups in cellulose may occur in more than one way, depending on the form taken by the $-\text{NH}-\text{CO}-$, for example—



It is certain that these two structures will have different energies, and hence the affinity will depend upon which structure predominates. Secondly, there is the simpler picture in which the substituents merely change the electron density about the amido group. To account for the increased affinity due to both electron-attracting and electron-repelling substituents, it is necessary in this case to postulate a single hydrogen bond between dye and fibre, which can be of the form—



and similarly for the iminolised form. Thus in one case the bonding hydrogen atom is contributed by the dye, and in the other by the fibre. Unfortunately, there is no experimental evidence available to show which of the above effects is the true one, and in point of fact it is highly probable that all contribute to some extent.

Another method of modifying the affinity of 1-benzamidoanthraquinone is by the introduction of substituents into the anthraquinone nucleus (Table IV). Here, again, the mechanisms discussed above for side-chain substitution are still applicable.

TABLE IV
Affinities of Substituted
1-Benzamidoanthraquinones
 $R^1-C_{14}H_9O_2-NH-CO-C_6H_4-R^2$

Dye	Mol. Wt.	Affinity, $-\Delta\mu^\circ$ (kcal./mole)
1-Benzamidoanthraquinone ...	327	2.37
1-Benzamido-4-methoxyanthraquinone ...	357	3.59
1-Benzamido-5-methoxyanthraquinone ...	357	3.59
1-p-Chlorobenzamidoanthraquinone ...	361.5	3.02
1-p-Chlorobenzamido-5-methoxyanthraquinone ...	391.5	4.06

So far, only changes in the dye molecule which lead to increased affinity have been discussed; but of equal interest from the point of view of mode of attachment are those changes which have the reverse effect. In the acylaminoanthraquinones there are three molecular modifications which result in reduced affinity, and these will be considered separately. Firstly, when the hydrogen atom of the amido group is removed by methylation, as in 1-N-methylacetamidoanthraquinone, there is a considerable reduction in affinity. Again, probably no single factor is contributing to this loss of affinity. The removal of the amido hydrogen atom means that only the hydrogen atom of a cellulose molecule is available to form bonds between dye and fibre. At the same time any iminisation of the amido group is prevented, so that the length of conjugated chain in the molecule is reduced. Also, the steric interaction of the N-methyl with the O- group will twist the side-chain out of the plane of the anthraquinone nucleus, thereby reducing the ease of adlineation of the molecule along the fibre, hindering the formation of hydrogen bonds and reducing the van der Waals attraction. A very marked decrease in affinity is observed when a methylene group is introduced between the amido group and the phenyl ring in 1-benzamidoanthraquinone (cf. 1-phenylacetamidoanthraquinone). Similar effects have been observed in a series of azoic coupling components by Krzikalla and Eistert¹⁰, and are

presumably due to the methylene group forming a complete break in any possible conjugated chain between side-chain and nucleus. Thirdly, the introduction of a substituent into the *ortho* position of the benzene ring of 1-benzamidoanthraquinone results in a decrease in affinity of approx. 0.3 kcal./mole (Table III). Here similar arguments again apply, the steric action of the *ortho* substituent causing the benzene nucleus to be out of the plane of the rest of the molecule, thereby reducing the amount of hydrogen bonding and also the area available for van der Waals attractive forces to be operative.

Summarising the results on the monosubstituted anthraquinones, it appears to be advantageous to have—

- A planar molecule
- Maximum conjugation for greatest possible van der Waals attraction
- An amido or an amino group capable of forming hydrogen bonds with the fibre.

There is no evidence to indicate that solubility in the aqueous phase plays a major rôle in determining the differences in affinity between dyes.

(b) Disubstituted Anthraquinones

The compounds which have been examined under this heading are those containing groups, such as amino and benzamido, which are capable of forming hydrogen bonds with the fibre. In general, all the observations made on the monosubstituted anthraquinones hold for these disubstituted derivatives. The effect of the second group is to increase the affinity, but the amount of the increase depends also on the relative positions of the two groups.

For the diamino compounds the affinity must depend on the ability of the amino groups to form hydrogen bonds with the fibre, and it appears that the distance apart of these groups is of importance. Thus the 1:8 and 1:4 derivatives, with a spacing of approx. 5 Å., have low affinities; while 1:5-diaminoanthraquinone, in which the spacing is approx. 9 Å., has a much greater affinity, presumably because the distance apart of the amino groups is approaching that of the repeat spacing in cellulose.

TABLE V

Dye	Extinction Coefficient	Affinity, $-\Delta\mu^\circ$ (kcal./mole)
1-Aminoanthraquinone ...	—	< 1.00
1:4-Diaminoanthraquinone ...	—	1.46
1:5-Diaminoanthraquinone ...	—	2.31
1:8-Diaminoanthraquinone ...	—	1.49
1-Benzamidoanthraquinone ...	5,730	2.37
1:4-Dibenzamidoanthraquinone ...	9,790	4.29
1:5-Dibenzamidoanthraquinone ...	8,570	3.57
1:8-Dibenzamidoanthraquinone ...	6,770	2.58
1:4:5-Tribenzamidoanthraquinone ...	10,100	4.36

For the benzamido derivatives, however, the positional differences can be explained by considering the conjugation in the molecule.

The 1:8 derivative has an affinity only slightly higher than that of 1-benzamidoanthraquinone. In this molecule it is impossible for any one conjugated chain to include both substituents, and hence the length of the conjugation is the same as for the monosubstituted compound. In 1:5-dibenzamidoanthraquinone conjugation can extend all over the molecule, but the resulting chain is not straight; whereas in the 1:4 derivative a practically linear system of single and double bonds is possible. It would appear that not only the length of conjugation is important in these dyes but also its linearity.

For a related series of compounds, such as the benzamidoanthraquinones, the conjugation in the molecule may be expected to be related to the absorption spectra. It has been found possible¹¹ to calculate approximate extinction coefficients of some simple aromatic and aliphatic compounds by assuming an effective area for the π electrons in the molecule, and from this calculating the light absorbed on the basis of the area of the light beam obscured by the π electrons. Thus, in a related

series of compounds, where it is possible to assume an equal probability of electronic transitions, the extinction coefficients of the band of longest wavelength should be proportional to the effective area of π electrons in the molecule, which in turn is the area available for van der Waals attraction. On these grounds, assuming a constant amount of hydrogen bonding throughout the series, the affinities should run parallel to the extinction coefficients. This is shown in Fig. 2 to be true of the benzamidoanthraquinones. Again, the dye might be expected to lie with its colour axis for the band of longest wavelength along the fibre. Dichroic measurements confirm this¹².

Superimposed on these van der Waals attractions there must also be the dipolar or hydrogen-bonding forces. These, as in the monosubstituted derivatives, are again modified by introducing groups with no affinity for cellulose into the *meta* and *para* positions of the benzene side-chains (Table I). Substitution in the *ortho* position again reduces the affinity, while replacement of the amido hydrogen atom by methyl depresses it to a very small value (e.g. in 1:4-bis-*N*-methylbenzamidoanthraquinone).

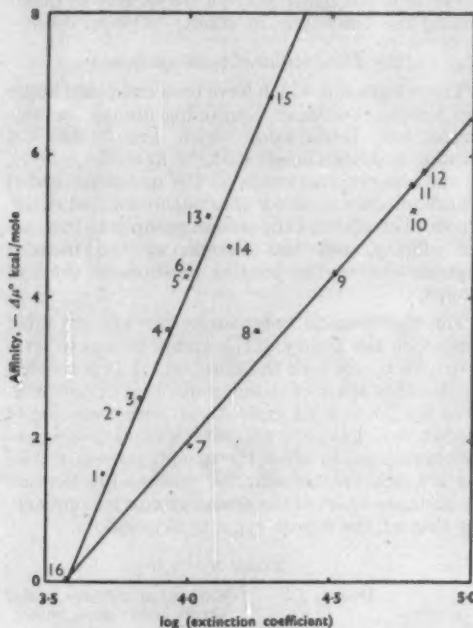
Summarising, the results show that the disubstituted compounds behave in the same way as the monosubstituted ones, with the proviso that the relative positions of the groups must be taken into consideration.

(c) Other Compounds

The only benzamido compound to be examined under this heading is 1:4:5-tribenzamidoanthraquinone, which has an affinity little different from that of the 1:4-dibenzamidoanthraquinone (Table V). This is in agreement with the suggestion that maximum conjugation occurs between the 1:4 positions, which will thus predominate over conjugation between the 1:5 and between the 4:5 positions. Its affinity, again, relates to the logarithm of the extinction coefficient of the band of longest wavelength (Fig. 2).

Finally, for substituted anthraquinone compounds, it must be noted that the distance between the two groups having hydrogen-bond-forming capabilities in derivatives of the dinuclear aliphatic bis-carboxamide type (Table II) does not seem to affect the affinity. This is in contrast to the direct cotton dyes, in which a spacing of 10.3 Å. appears to be advantageous.

The remaining compounds to be considered are a number of carbocyclic vat dyes containing no substituents whatever (Table VI). It can be seen that the affinity increases with the number of condensed rings in the dye. These results can be explained on the basis of non-polar bonding forces, a fact which is confirmed by the large affinity which leuco Caledon Jade Green XN has for activated carbon (unpublished work by Weston). It is interesting, too, that the leuco dyes exhibit a dichroism of the same magnitude as Congo Red, being aligned with their colour axis parallel to that of the fibre¹³. Hence it must be assumed that the colour and the affinity are determined by the same part of the molecule,



1. 1-Acetamidoanthraquinone
2. 1-Benzamidoanthraquinone
3. 1:5-Dibenzamidoanthraquinone
4. 1:5-Dibenzamidoanthraquinone
5. 1:4-Dibenzamidoanthraquinone
6. 1:4:5-Tribenzamidoanthraquinone
7. 1:2'-Benzanthraquinone
8. Anthranthrene
9. 1:2':6':7'-Dibenzopyrene-7:14-quinone
10. 1:5-Dibenzanthrone
11. Dibenzanthrone
12. Pyranthrene
13. 1:5-Phthaloylbis-1-anthraquinonylamide
14. Caledon Red BN
15. Indanthrone (Simons, unpublished work)
16. Aethranthone

FIG. 2

which is to be expected, since the colour is related to the number of π electrons available, and so will be the van der Waals bonding forces. Just as for the benzamidoanthraquinones, the affinities fall in the same order as the extinction coefficients (Fig. 2). A comparison between the two lines for the carbocyclic and the anthraquinone dyes shows that both pass through the point for anthraquinone. The anthraquinone derivatives show the larger slope, which is probably due to the enhancement of their affinity by polar hydrogen-bonding forces, which are lacking in the carbocyclic compounds.

TABLE VI

Dye	Extinction Coefficient	Affinity, $-\Delta\mu^\circ$ (kcal. per mole)	No. of Condensed Nuclei
1':2'-Benzanthraquinone ...	10,300	1.91	4
Anthanthrone ...	17,500	3.53	6
1':2':6':7'-Dibenzo-pyrene-7:14-quinone	31,300	4.35	6
isoDibenzanthrone ...	62,300	5.20	9
Pyranthone ...	60,000	5.50	8
Dibenzanthrone ...	67,100	5.77	9

CONCLUSIONS

The results discussed above strongly indicate that the affinity of vat dyes arises from two major types of attachment to the fibre, hydrogen bonds and van der Waals attractive forces. In order that either or both of these bonds should be formed to a maximum extent the molecule should—

- Be planar to allow best contact with the fibre
- Have maximum conjugation for greatest possible van der Waals attraction
- Have a group capable of forming hydrogen bonds with the fibre.

No evidence was found to show that solubility in the aqueous phase plays a major part in determining affinity.

The importance of the benzamido group in anthraquinone vat dyes appears to lie in the fact that a simple hydrogen-atom tautomerism can bring the side-chain into conjugation with the nucleus, thereby increasing the length of the conjugated chain. Also, by suitable substitution in the benzene ring, the hydrogen-bonding capabilities of the amido group can be enhanced.

Generally, in compounds containing two hydrogen-bonding groups, their distance apart is unimportant, but in the majority of these compounds the molecule is of such a labile structure that any definite conclusion is impossible.

Of interest is the correlation obtained between affinity and light absorption, which emphasises the contribution to the affinity of van der Waals forces from the π electrons.

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Appendix

Dye	Mol. Wt.	Concn. of Dye on Fibre $\times 10^3$ g.-ion/kg.	Final Concn. of Dyebath (g.-ion/litre) $\times 10^3$	[Na] _f (g.-ion/litre) $\times 10^3$	Affinity $-\Delta\mu^\circ$ (kcal. per mole)
Anthraquinone (I)* ...	208	Trace	1.503	1.079	<1
1-Chloroanthraquinone ...	242.5	Trace	1.289	1.079	<1
1-Methylantraquinone ...	222	Trace	1.408	1.079	<1
1-Methoxyanthraquinone ...	238	Trace	1.313	1.079	<1
1-Aminoanthraquinone (XII) ...	223	Trace	1.402	1.079	<1
2-Aminoanthraquinone ...	223	Trace	1.402	1.079	<1
1-Acetamidoanthraquinone ...	265	2.65	4.08	1.176	1.52
2-Acetamidoanthraquinone ...	265	2.65	4.08	1.176	1.53
1-Chloroacetamidoanthraquinone ...	299.5	2.1	3.23	1.040	1.46
1-N-Methylaminoanthraquinone ...	237	2.30	3.54	1.136	1.46
1-N-Methylacetamidoanthraquinone ...	279	Trace	1.120	1.079	<1
1-Phenylacetamidoanthraquinone ...	341	Trace	0.916	1.079	<1
1-op-Dimethylphenylaminoanthraquinone...	327	4.57	7.03	0.950	2.00
1:8-Diaminoanthraquinone ...	238	2.8	4.31	1.309	1.49
1:4-Diaminoanthraquinone ...	238	2.68	4.12	1.309	1.46
1:5-Diaminoanthraquinone ...	238	10.10	15.53	1.299	2.31
1:4-Bis-N-methylaminoanthraquinone ...	266	2.75	4.23	1.171	1.55
1-Benzylaminoanthraquinone ...	313	Trace	1.045	1.079	<1

*Roman numerals refer to the formulae on pp. 130 and 131.

Dye	Mol. Wt.	Concn. of Dye on Fibre $\times 10^3$		Final Concn. of Dyebath (g.-ion/litre) $\times 10^3$	[Na] (g.-ion/litre) $\times 10^3$	Affinity $-\Delta\mu^\circ$ (kcal. per mole)
1-Benzamidoanthraquinone (XIII)	327	8.09	12.44	0.945	1.093	2.37
2-Benzamidoanthraquinone	327	4.97	7.64	0.940	1.087	2.05
1- β -Naphthoamidoanthraquinone	377	43.8	67.36	0.767	1.149	3.61
1-Amino-4-benzamidoanthraquinone	342	18.56	28.54	0.888	1.108	2.94
1-Benzamido-4-methoxyanthraquinone	357	45.0	69.2	0.812	1.151	3.59
1-Amino-5-benzamidoanthraquinone	342	11.20	12.23	0.898	1.092	2.39
1-Benzamido-5-methoxyanthraquinone	357	44.6	68.6	0.812	1.150	3.59
1- <i>p</i> -Chlorobenzamido-5-methoxyanthraquinone	391	74.7	114.9	0.693	1.202	4.06
1- <i>o</i> -Chlorobenzamidoanthraquinone	361.5	4.3	6.13	0.856	1.086	1.98
1- <i>m</i> -Chlorobenzamidoanthraquinone	361.5	12.9	19.84	0.847	1.100	2.73
1- <i>p</i> -Chlorobenzamidoanthraquinone	361.5	19.58	30.51	0.837	1.110	3.02
1- <i>o</i> -Methylbenzamidoanthraquinone	341	4.9	7.54	0.909	1.087	2.07
1- <i>m</i> -Methylbenzamidoanthraquinone	341	13.8	21.22	0.897	1.101	2.74
1- <i>p</i> -Methylbenzamidoanthraquinone	341	12.6	19.38	0.898	1.099	2.68
1- <i>p</i> -Methoxybenzamidoanthraquinone	357	16.7	25.68	0.851	1.105	2.90
1:4-Dibenzamidoanthraquinone (XIV)	446	87.2	134.1	0.578	1.224	4.29
1:4-Bis- <i>o</i> -chlorobenzamidoanthraquinone	515	25.5	39.22	0.571	1.119	3.42
1:4-Bis- <i>m</i> -chlorobenzamidoanthraquinone	515	151	232.2	0.394	1.338	4.98
1:4-Bis- <i>p</i> -chlorobenzamidoanthraquinone	515	179	275.3	0.355	1.390	5.20
1:4-Bis- <i>o</i> -methylbenzamidoanthraquinone	474	38.0	58.44	0.605	1.139	3.66
1:4-Bis- <i>m</i> -methylbenzamidoanthraquinone	474	110	169.2	0.504	1.264	4.56
1:4-Bis- <i>p</i> -methylbenzamidoanthraquinone	474	111	170.7	0.503	1.268	4.57
1:4-Bis- <i>p</i> -methoxybenzamidoanthraquinone	506	92	141.4	0.488	1.233	4.44
1:4-Bis- <i>o</i> -bromobenzamidoanthraquinone	604	25.3	39.91	0.482	1.120	3.54
1:4-Bis- <i>N</i> -methylbenzamidoanthraquinone	474	Trace		0.659	1.079	<1
1:5-Dibenzamidoanthraquinone	446	35.7	54.91	0.651	1.136	3.57
1:5-Bis- <i>o</i> -chlorobenzamidoanthraquinone	515	8.6	13.23	0.595	1.093	2.69
1:5-Bis- <i>m</i> -chlorobenzamidoanthraquinone	515	67.5	103.8	0.512	1.190	4.17
1:5-Bis- <i>p</i> -chlorobenzamidoanthraquinone	515	84.5	130.0	0.488	1.220	4.37
1:5-Bis- <i>o</i> -methylbenzamidoanthraquinone	474	9.6	14.76	0.645	1.095	2.71
1:5-Bis- <i>m</i> -methylbenzamidoanthraquinone	474	44.3	68.13	0.597	1.150	3.77
1:5-Bis- <i>p</i> -methylbenzamidoanthraquinone	474	42.1	64.75	0.600	1.146	3.73
1:5-Bis- <i>o</i> -methoxybenzamidoanthraquinone	506	5.5	8.46	0.610	1.088	2.39
1:5-Bis- <i>m</i> -methoxybenzamidoanthraquinone	506	46.6	71.67	0.552	1.153	3.86
1:5-Bis- <i>p</i> -methoxybenzamidoanthraquinone	506	63.8	98.12	0.528	1.182	4.11
Succinylbis-1-anthraquinonylamide	528	9.7	14.92	0.578	1.110	3.59
Glutarylbis-1-anthraquinonylamide	542	10.1	15.53	0.563	1.111	3.63
Adipylbis-1-anthraquinonylamide	556	10.0	15.38	0.548	1.111	3.64
Pimelylbis-1-anthraquinonylamide	570	7.5	11.54	0.537	1.106	3.46
Suberylbis-1-anthraquinonylamide	584	7.4	11.38	0.525	1.103	3.46
Azelaylbis-1-anthraquinonylamide	598	5.6	8.61	0.515	1.096	3.28
Sebacylbis-1-anthraquinonylamide	612	7.8	12.00	0.500	1.104	3.53
isoPhthaloylbis-1-anthraquinonylamide	576	54.0	83.05	0.467	1.258	5.10
1:4:5-Tribenzamidoanthraquinone	565	77.4	119.0	0.444	1.437	4.56
Anthanthrone (VI)	306	47.4	72.9	0.954	1.155	3.53
Dibenzanthrone (IX)	456	429	659.8	0.647	1.925	5.77
isoDibenzanthrone (X)	456	265	407.6	0.662	1.560	5.20
1':2'-Benzanthraquinone (II)	258	5.0	7.09	1.205	1.087	1.91
Pyranthrone (V)	406	392	602.9	0.735	1.842	5.59
1':2':6':7'-Dibenzopyrene-7:14-quinone (IV)	332	118	181.5	0.775	1.280	4.35
3-Benzamido-2-naphthol	263	5.45	8.38	1.180	1.085*	1.65
2-Hydroxy-3-naphthoanilide	263	7.6	11.69	1.177	1.086*	1.88
3-Amino-2-naphthol	159	Trace		1.966	1.079*	<1
3-Acetamido-2-naphthol	201	Trace		1.555	1.079*	<1
Caledon Red BN (C.I. 1162)...	375	151.6	233.2	0.621	1.340†	4.70

*Taken as monobasic

†Taken as dibasic

The Dyeing of Acetate Rayon with Disperse Dyes

III—The Influence of Dispersing Agents on the Rate of Dyeing

C. L. BIRD, Miss P. HARRIS, and F. MANCHESTER

It is shown that the dispersing agents added to disperse dyebaths stabilise the dye suspension and act as restraining and retarding agents. With some dyes, especially at low temperatures, the normal retarding effect is reversed, probably owing to an increase in the rate of dissolution of crystalline particles of dye in presence of dispersing agent.

INTRODUCTION

During the course of an investigation which was mainly concerned with the use of disperse dyes in dyeing acetate rayon packages, Corbière¹ observed that the rate of dyeing of acetate rayon hanks was increased by adding certain dispersing agents to the dyebath. Corbière's dyeings were carried out for times up to 2 hr., using two dyes, viz. Cibacet Sapphire Blue G (Ciba), dyed at 50° and 80°C., and Acetoquinone Rose N (Fran), dyed at 50°C. Additions to the dyebath, per litre, were as follows—(1) 0.5 g. soap, (2) 2.0 g. soap, (3) 0.5 g. soap + 2.0 g. Albatex PO (Ciba), and (4) 0.5 g. soap + 2.0 g. "Product B". On plotting adsorption-*versus*-time curves it was seen that the additional dispersing agent had increased the rate of dyeing in each case, especially at the lower temperature, and "Product B" was particularly effective. "Product B" is similar to Emulphor EL (FBy), which is a condensate of 1 mol. of castor oil with 40 mol. of ethylene oxide² and is a very good solubilising agent for disperse dyes³.

Bird³ has shown that the dispersing agents added to disperse dyebaths exert a restraining effect, i.e. that the percentage exhaustion of dye at equilibrium is decreased, a fact which can be attributed to competition between the fibre and the dispersing agent for the available dye. This behaviour is similar to that described by Speke⁴ in connection with leuco vat dyes and the dispersing agent Dispersol VL (ICI). Speke showed that this compound is a retarding agent for vat dyes, as well as a restraining agent; i.e. it decreases the rate of dyeing. A similar retarding effect might be expected in disperse dyebaths.

We have repeated some of Corbière's experiments with Cibacet Sapphire Blue G. Whilst no increase in rate of dyeing was observed at 80°C., there was a significant increase at 50°C. when a dyebath containing only 0.5 g. soap per litre was compared with a dyebath containing 0.5 g. soap and 2.0 g. Emulphor EL per litre. The results at 50°C. are given in Table I.

TABLE I
Dyeings with Cibacet Sapphire Blue G
(2% Dye. Liquor : yarn ratio 30 : 1.
Temp. 50°C. Time 15 min.)

Dispersing Agent (g./litre)	Exhaustion (%)
Soap (0.5)	20
Soap (2.0)	26
Soap (0.5) + Albatex PO (2.0) ...	29
Soap (0.5) + Emulphor EL (2.0) ...	41

In view of these apparently conflicting results, it was decided to investigate the problem in more detail. The following dyes, having relatively very low, medium, and high aqueous solubility, respectively, at 80°C.⁵, were chosen—

Duranol Blue 2G 300 (ICI)

Dispersol Fast Scarlet B 150 (ICI)

Serisol Fast Red 2RD (YDC).

These dyes were applied to acetate rayon at 80°C. for varying times in presence of (a) 1% Lissapol LS (ICI), and (b) sufficient Lissapol LS to give approx. 50% exhaustion at equilibrium (see Fig. 1), the rate of dyeing being expressed in terms of the time of half-dyeing, i.e. the time taken to reach half the equilibrium exhaustion.

The reason for the choice of sufficient Lissapol LS to give 50% exhaustion was the belief that the resulting rates of dyeing might be comparable with the rates of diffusion in the fibre. The results (Table II) suggest that this view is untenable. We have not yet determined the rate of diffusion of Serisol Fast Red 2RD in cellulose acetate, but, since this dye is rapid dyeing, it is very unlikely to diffuse at a slower rate than Duranol Blue 2G, which is known⁵ to diffuse extremely slowly.

The results for Duranol Blue 2G are illustrated in Fig. 2, which also includes an isotherm obtained with 5 g. Lissapol LS per litre. The times of half-dyeing obtained from curves for all three dyes are given in Table II.

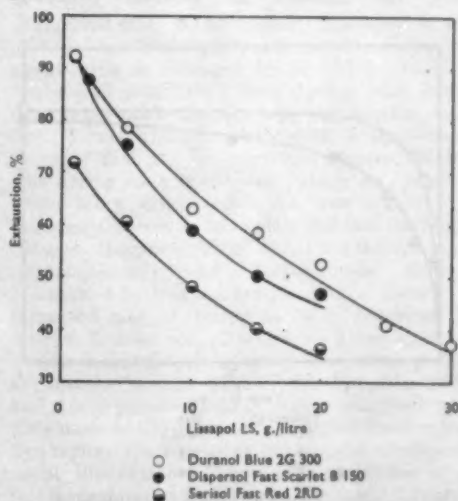


FIG. 1—Restraining Effect of Lissapol LS

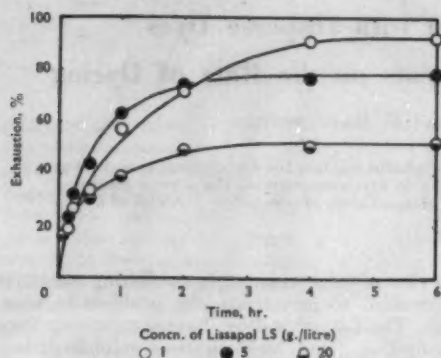


FIG. 2—Effect of Increasing Amounts of Lissapol LS on Rate of Dyeing of Duranol Blue 2G 300 at 80°C.

Since the exhaustion at equilibrium varied from 50 to 90%, a possible cause of the variation in the time of half-dyeing was the difference in the amount of dye available to the fibre. Further dyeings with Dispersol Fast Scarlet B and Serisol Fast Red 2RD were therefore carried out, using 1 g. Lissapol LS per litre and sufficient dye to give an equilibrium exhaustion of approx. 1.8 mg. per gram of yarn. The results are given in Table III, from which it is seen that the time of half-dyeing is not greatly influenced by fairly large variations in the amount of dye available to the fibre.

On the other hand, when the percentage exhaustion at equilibrium is kept constant by varying the amount of dye, increasing the amount of dispersing agent still decreases the rate of dyeing with Dispersol Fast Scarlet B and Serisol Fast Red 2RD, as illustrated in Fig. 3.

In a second series of experiments, using the same dyeing apparatus, purified dye was applied to acetate rayon for short periods in presence of varying amounts of three purified dispersing agents, viz. Lissapol LS (sodium 1-oleyl-4-

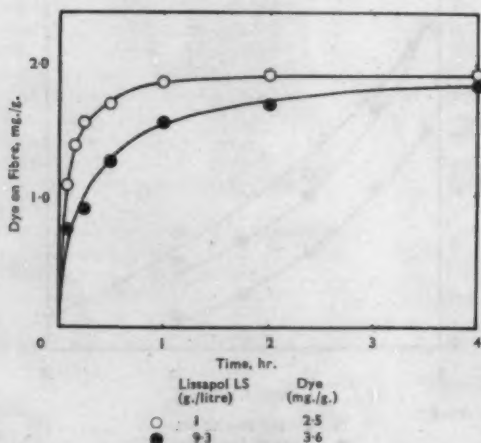


FIG. 3—Retarding Effect of Lissapol LS on Dyeing of Serisol Fast Red 2RD at 80°C.

anisidine-2-sulphonate), Lissapol C (sodium cetyl-oleyl sulphate), and a mixture of sodium *sec.*-alkyl(C_{10} - C_{18}) sulphates. The dyes used were *p*-nitroaniline→diethylaniline, applied for 10 min. at 60°C., and *p*-nitroaniline→*N*-ethyl-*N*-β-hydroxyethyl-aniline (Dispersol Fast Scarlet B), applied for 15 min. at 20°C. and for 5 min. at 40°C. with addition of purified Lissapol LS only. The dyeing times were chosen after preliminary experiments to determine the optimum conditions for observing the increased rate of dyeing. *p*-Nitroaniline→diethylaniline has an aqueous solubility of only 0.2 mg./litre at 80°C., but Dispersol Fast Scarlet B is much more soluble (7 mg./litre) and does not show the increased rate of dyeing until the temperature is lowered to 20°C., when the aqueous solubility is reduced to <0.2 mg./litre. The results are illustrated in Fig. 4 and 5.

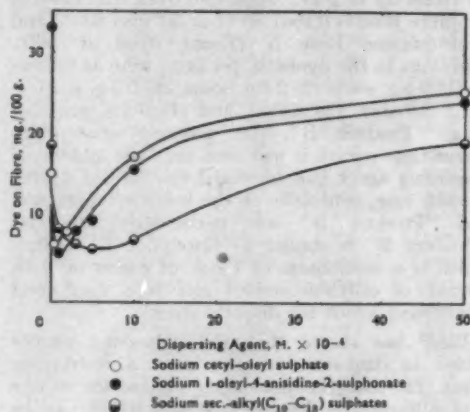


FIG. 4—Effect of Dispersing Agent in 10-min. Dyeings of *p*-Nitroaniline→Diethylaniline at 60°C.

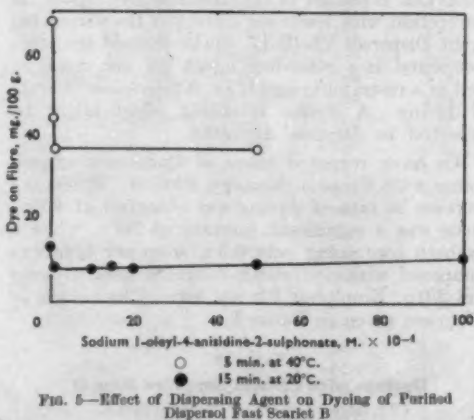


FIG. 5—Effect of Dispersing Agent on Dyeing of Purified Dispersol Fast Scarlet B

EXPERIMENTAL

The percentage purity of each commercial dye was determined by colorimetric comparison against a sample of purified dye, and the amounts of commercial dye were adjusted so that each dyebath

TABLE II

Effect of Increasing Amounts of Lissapol LS on Time of Half-dyeing at 80°C.

Lissapol LS (g./litre)	Duranol Blue 2G Time of Half- dyeing (min.)	Exhaust- ion (%)	Dispersol Fast Scarlet B Time of Half- dyeing (min.)	Exhaust- ion (%)	Serisol Fast Red 2RD Time of Half- dyeing (min.)	Exhaust- ion (%)
1	45	92	4.5	90	4	74
5	20	78	—	—	—	—
9.3*	—	—	—	—	14	50
15*	—	—	7	50.5	—	—
20*	12	51	—	—	—	—

*Sufficient to give approx. 50% exhaustion at equilibrium.

TABLE III

Effect of Varying the Amount of Dye on Time of Half-dyeing at 80°C.
(1 g. Lissapol LS per litre)

Initial Concn. of Dye (mg./g.)	Dispersol Fast Scarlet B Time of Half-dyeing (min.)	Dye on Fibre at Equilibrium (mg./g.)	Serisol Fast Red 2RD Time of Half-dyeing (min.)	Dye on Fibre at Equilibrium (mg./g.)
3.6	4.5	3.2	4	2.8
2.5	—	—	4.5	1.9
1.9	4	1.8	—	—

contained the equivalent of 3.6 mg. pure dye per gram of yarn. With Dispersol Fast Scarlet B this amount corresponds to a 1.5% commercial dyeing.

Dyeings on solvent-extracted Celanese secondary acetate yarn were carried out in a modified Marney machine, as described in Part I². Dyeings for 10 min. or less were done in triplicate; other dyeings were in duplicate. When dyeing for longer than 2 hr., the tubes were fitted with small water-cooled condensers to avoid concentration of the dye liquor. Estimation of dye on the fibre and in the residual dyebath was carried out in the usual way using a Spekker absorptiometer (Hilger & Watts).

Suspensions of the purified dyes were prepared by grinding in a mortar with a minimum amount of dispersing agent dissolved in water, the amounts used being negligible compared with the dyebath additions which were made later. After diluting the resulting dispersion with water to 100 ml. in a stoppered cylinder, the suspension was allowed to stand overnight, when the supernatant liquor was withdrawn and diluted to give a concentration of 2 g. dye per litre. Suspensions prepared in this way had a maximum particle size of 8–10 μ ., with the majority of the particles around 1 μ ., although no particle size analysis was carried out.

The cellulose acetate yarn (1 g.) was packed loosely into the cage, which was then agitated for 5 min. at the temperature of the experiment in 70 ml. of water containing the particular amount of dispersing agent. The yarn was then raised, and 10 ml. of the dye suspension added; after vigorous stirring, the yarn was returned and dyeing continued for the required period. At the end of this time the cage was removed and agitated in two successive 250-ml. lots of cold water. The yarn

was then removed from the cage, squeezed between filter papers, and dissolved in acetone. The amount of dye present was calculated on the original conditioned weight of yarn.

DISCUSSION

It is seen from Table II that increasing the amount of Lissapol LS increases the rate of dyeing with Duranol Blue 2G, but with the other two dyes the rate of dyeing is decreased, especially with Serisol Fast Red 2RD. These results are illustrated in Fig. 2 by the 1 g./litre and 5 g./litre Lissapol LS curves, and by Fig. 3.

Duranol Blue 2G is virtually insoluble in water at 80°C., but it is slightly soluble when a dispersing agent such as Lissapol LS is added. It is now fairly well established that dyeing with disperse dyes takes place through aqueous solution, and at first it was thought that, with a dye such as Duranol Blue 2G, the very low aqueous solubility was acting as a bottleneck, which was overcome when more dispersing agent was added. It is necessary, however, to explain the fact that, whilst Cibacet Sapphire Blue G (1:4:5:8-tetra-amino-anthraquinone) and Acetoquinone Rose N (1-amino-4-hydroxyanthraquinone) show the increased rate of dyeing at 50°C., Dispersol Fast Scarlet B does not, although all three dyes have similar aqueous solubilities². The most probable explanation is that, with Cibacet Sapphire Blue G and Acetoquinone Rose N, dye is adsorbed by the fibre more quickly than dye particles dissolve in the dye liquor. On increasing the amount of dispersing agent dissolution of crystalline particles of dye takes place more rapidly and the rate of dyeing is increased.

TABLE IV

Relation between Solubilising Power and Rate of Dyeing of *p*-Nitroaniline \rightarrow Diethylaniline

Dispersing Agent	Solubility of Dye in 0.002 M. Dispersing Agent at 20°C. (mg./litre)	Concn. (M.) of Dis- persing Agent required to give 15 mg. Dye per 100 g. Yarn in 10 min. at 60°C.
Sodium octyl-oleyl sulphate ...	40	0.0007
Sodium 1-oleyl-4-anisidine-2- sulphonate	5.2	0.001
Sodium <i>sec.</i> -alkyl(C ₁₀ –C ₁₈) sulphates	1.2	0.003

With disperse dyes which dissolve relatively readily in hot water, e.g. those containing an *N*- β -hydroxyalkyl group, the normal retarding effect will be observed when increased amounts of dispersing agent are added to the dyebath, except at low temperatures.

The retarding effect of dispersing agents can be explained as follows. Dyeing of acetate rayon takes place from a dilute saturated aqueous solution which is in equilibrium with a saturated film of dye on the surface of the fibres. The solution is maintained saturated by almost instantaneous dissolution of suspended dye, and as long as the solution is saturated the concentration of dye in the surface film will not change. When an appreciable amount of dispersing agent is present, however, e.g. sufficient to reduce the equilibrium exhaustion to 50%, the whole of the suspended dye will be dissolved at a very early stage in the dyeing. This will not immediately affect the concentration of dye in the surface film, however, since work in progress has shown that, over a limited range, the saturation value is independent of the concentration of dispersing agent⁵. As dye now diffuses into the fibre, more dye will be taken up from the dyebath, which will no longer be saturated. Hence the concentration of dye in the surface film will decrease, and with it the rate of diffusion in the fibre and the overall rate of dyeing.

The second series of experiments (Fig. 4 and 5), besides illustrating the increased rate of dyeing, show that, on adding small amounts of dispersing agent, there is a rapid fall in the amount of dye taken up by the fibre in the early stages of dyeing. In the virtual absence of dispersing agent the

suspension is unstable, dye being immediately adsorbed on to the fibre or on to any glass surface in contact with it. On adding a little dispersing agent, which is adsorbed both by the fibre and by the very fine particles of dye, the rapid initial adsorption of dye by the fibre is eliminated.

At the points in Fig. 4 of minimum rate of dyeing, which is approximately the same for all three dispersing agents, the governing factor is the rate of dissolution of solid dye. This rate is apparently increased on adding more dispersing agent, as shown by the right-hand portions of the curves. The efficiency of the three dispersing agents in respect of their ability to increase the rate of dyeing is proportional to their solubilising powers, which are given in Table IV.

With Dispersol Fast Scarlet B (Fig. 5), rate of dissolution of solid dye is generally adequate, and in consequence this dye does not show the increased rate of dyeing above room temperature.

* * *

We desire to express our gratitude to Courtaulds' Scientific and Educational Trust Fund for a scholarship which enabled one of us (P.H.) to take part in this work.

DEPARTMENT OF COLOUR CHEMISTRY AND DYEING
THE UNIVERSITY
LEEDS 2

(Received 21st September 1954)

References

- ¹ Corbière, *Teintex*, **13**, 433 (1948).
- ² B.I.O.S. Report No. 421 (H.M. Stationery Office, 1946).
- ³ Bird, *J.S.D.C.*, **70**, 68 (1954).
- ⁴ Speke, *ibid.*, **66**, 569 (1950).
- ⁵ Manchester, *Ph.D. Thesis* (University of Leeds, 1954).

CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

Dry Cleaning of Cellulose Triacetate

The introduction of a new textile fibre, cellulose triacetate, has recently been announced¹. It is claimed that this fibre possesses certain advantages over normal (secondary) acetate rayon, notably higher melting point, lower water imbibition, and high resistance to chemicals and weathering, which should make it a useful addition to the available textile materials. Cellulose triacetate can be dyed with disperse dyes, but it has been found that the fastness of such dyeings to dry cleaning in trichloroethylene is extremely poor. Cellulose triacetate dyed with some forty individual dyes has so far been examined, and in every case there was serious bleeding of dye into the solvent and transfer on to adjacent undyed patterns. Furthermore, appreciable creasing and shrinkage of both woven and knitted triacetate fabrics have been observed on cleaning in this solvent. These defects are attributed to swelling of the fibre by trichloroethylene.

The fastness of cellulose triacetate dyed with disperse dyes to dry cleaning in perchloroethylene and white spirit has been found to be satisfactory with the majority of dyes so far examined, and

there appears to be no appreciable swelling, creasing, or shrinkage of the fibre on treatment in these solvents.

It is apparent, therefore, that it will be impossible to clean satisfactorily garments made from cellulose triacetate in trichloroethylene, though cleaning in perchloroethylene and white spirit is not expected to cause undue difficulty.

In order that dry cleaners may be in a position to clean without impairing any garments submitted to them, the Dyers and Cleaners Research Organisation has recently advised its members who employ trichloroethylene as a dry-cleaning solvent to change over to the use of perchloroethylene, although the necessary plant adaptations and costs will entail inconvenience and hardship, and will take some time.

E. J. DAVIES
W. K. RHODES

DYERS AND CLEANERS RESEARCH ORGANISATION
4 LIFTON PLACE
LEEDS 2

18th January 1955

¹ Cf. J.S.D.C., 71, 105 (Feb. 1955).

Notes

**Meetings of Council and Committees
February**

Council—9th
Finance—9th and 15th
Publications—15th
Colour Index Editorial Panel—17th
Society's Medals—7th
Diplomas Executive Subcommittee—16th
Perkin Centenary Executive Committee—25th
Northern Ireland Symposium Papers Subcommittee—4th
Review of Textile Progress Committee—14th

Resignation of the Honorary Secretary

Council has learnt with great regret that, owing to increasing business commitments, Mr. John G. Hopkinson feels that he cannot devote the necessary time to the work of Honorary Secretary of the Society. He is therefore unable to accept nomination for the forthcoming session, but will continue in office until a successor has been nominated.

Fred Scholefield

M.Sc., F.R.I.C., F.T.I., F.S.D.C.

Honorary Member of the Society

Council unanimously resolved at its meeting held on 9th February 1955 that Mr. F. Scholefield be

made an Honorary Member of the Society in recognition of sustained and outstanding services to the Society.

Croyden Meredith Whittaker

D.Sc., F.T.I., F.S.D.C.

Honorary Member of the Society

Council unanimously resolved at its meeting held on 9th February 1955 that Dr. C. M. Whittaker be made an Honorary Member of the Society in recognition of sustained and outstanding services to the Society.

George Stuart James White

M.A., F.T.I., F.S.D.C.

Award of the Society's Gold Medal

On the recommendation of the Society's Medals Committee, Council has resolved that the Gold Medal of the Society be awarded to Mr. G. S. J. White for exceptional services to the Society and to the tinctorial and allied industries.

Clifford Collier Wilcock

A.R.T.C. (Salford), F.T.I., F.S.D.C.

Award of the Society's Silver Medal

On the recommendation of the Society's Medals Committee, Council has resolved that the Silver Medal of the Society be awarded to Mr. C. C. Wilcock for exceptional services to the Society and to the tinctorial and allied industries.

A.S.D.C. Examination

It is intended to hold an examination for the Associateship of the Society of Dyers and Colourists on Thursday-Saturday, 9-11th June 1955. Candidates are reminded that they must inform the Society by 31st March 1955 (or 16th April 1955 for overseas candidates) of the branch of tinctorial technology in which they wish to be examined.

Death

We regret to report the loss by death of Dr. E. A. Krähenbühl.

Royal Society National Committee for Chemistry

Mr. John Boulton, M.Sc., F.R.I.C., F.T.I., F.S.D.C., has accepted Council's invitation to represent the Society on the above body.

B.S. 1006 : 1955

At the meeting of Subcommittee ISO/TC 38/SC 1 held in Scarborough in June 1954¹ a test for Colour Fastness to Daylight was accepted.

In view of this, some slight alterations to B.S. 1006 : Part 1 : 1953² are necessary, and instead of issuing Amendment No. 3—which would in part countermand Amendment No. 2³—the Society's Light Fastness Subcommittee felt that a revised edition should be published, to be known as B.S. 1006 : 1955. The British Standards Institution are putting this in hand at once.

B.S. 1006 : 1955 will differ from the 1953 version in the following respects—

(1) The dyes used for the standards shall be—

1. Acilan Brilliant Blue FFR (FBy)
2. Acilan Brilliant Blue FFB (FBy)
3. Coomassie Brilliant Blue R (ICI)
4. Supramine Blue EG (FBy)
5. Solway Blue RN (ICI)
6. Alizarine Light Blue 4GL (S)
7. Soledon Blue 4BC (ICI)
8. Indigosol Blue AGG (DH).

(2) Exposure is now to Grades 4 and 3 on the Geometric Grey Scale instead of Grades 3 and 2.

(3) Assessments in the 7-8 region shall be made when a Grade 4 contrast has been produced on Standard 7 instead of Grade 3 as hitherto.

¹ J.S.D.C., 70, 294 (July 1954).

² B.S. 1006 : Part 1 : 1953 (London: British Standards Institution). Cf. J.S.D.C., 68, 197 (June 1952).

³ Amendments PD 1747 (Nov. 1953) and PD 1816 (Feb. 1954).

Terms and Definitions Committee

The Terms and Definitions Committee, in submitting the appended Tentative Definitions, wish to state that they have met twenty-one times, and have defined some 105 terms covering a wide range of subjects.

They consider that they have now reached the end of the first phase of their work, which should be consolidated by adopting their definitions,

amended where desirable after consideration of criticisms and suggestions from members of the Society.

The Committee thank all those who have been good enough to write to them regarding their work, and assure them that every suggestion will be carefully considered before the publication of "Recommended" definitions. They will welcome comments on and criticisms of the present list and of those given in previous numbers of the *Journal*—May, July, August 1952; January, April, July, November 1953; February 1954. These should be addressed to the Honorary Secretary of the Committee (S. R. Cockett Esq., c/o Messrs. Sandoz Products Ltd., Canal Road, Bradford 2, Yorkshire) to reach him by 30th April 1955.

Tentative Definitions

60. COMPATIBLE DYES

Dyes which, when mixed together, behave in dyeing as a homogeneous dye.

NOTE—Compatibility in dyeing is usually assessed by means of a series of dyeings in which the time of dyeing or the total quantity of dye applied to the fibre is varied. If the dyeings so produced are all of very similar hue, then the dyes in the mixture are said to be compatible. On occasion the term may be extended to include fastness properties, dyeings of compatible dyes remaining of the same, or nearly the same, hue when subjected to fading or washing tests.

61. MILLING ACID DYE

An acid dye having high fastness to wet processing on wool, and normally applied to protein fibres from weakly acid or neutral dyebaths.

62. AFTERCHROME PROCESS

A method of dyeing in which the fibre is dyed with a chrome dye and afterwards treated with a chromium compound to form a dye-chromium complex within the fibre.

63. ANTI-CHLOR

A chemical used to remove traces of residual active chlorine from materials.

64. BURL DYEING

The dyeing of wool piece-goods so as to colour any material present as an impurity.

65. CAKE

The package of filament yarn produced in the viscose spinning industry by means of the Topham box.

NOTE—In appearance it is almost cylindrical, and common dimensions are—height 3½ in., external diameter 6 in., internal diameter 4 in. It is not supported by any internal bobbin or tube.

66. CHROMATE DYEING PROCESS

A method of dyeing in which the fibre is treated in a dyebath containing a suitable chrome dye together with ammonium chromate, whereby a dye-chromium complex is formed within the fibre.

67. CHROME DYE

A dye which is capable of forming a chelate complex with a chromium atom.

68. CHROME MORDANT DYEING PROCESS

A method of dyeing wherein the fibre is pretreated in a solution of a chromium compound and subsequently dyed with a chrome dye to yield a dye-chromium complex within the fibre.

69. DRY CLEAN

To clean garments or fabrics by treating them in an organic solvent, as distinct from aqueous liquors. Examples of such solvents are white spirit, trichloroethylene, perchloroethylene.

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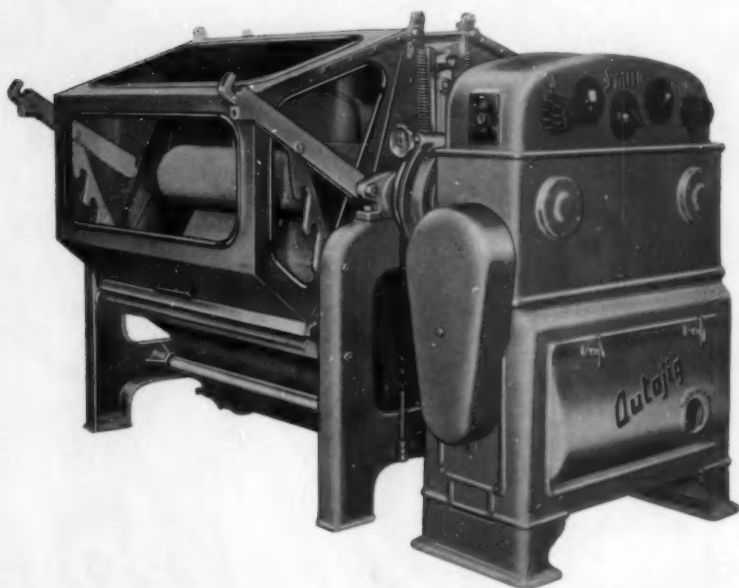
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An apt illustration of Hardy's dictum that "creeds are transient but human needs are eternal", the house has existed as an inn for over 550 years but the charterhouse itself is in ruins. The wool trade, too, continues to flourish, although methods have radically changed since the days when the dyers gathered the materials for their work from the fields and hedgerows. That part of dyeing at least, has been greatly simplified, thanks to such companies as Brotherton.

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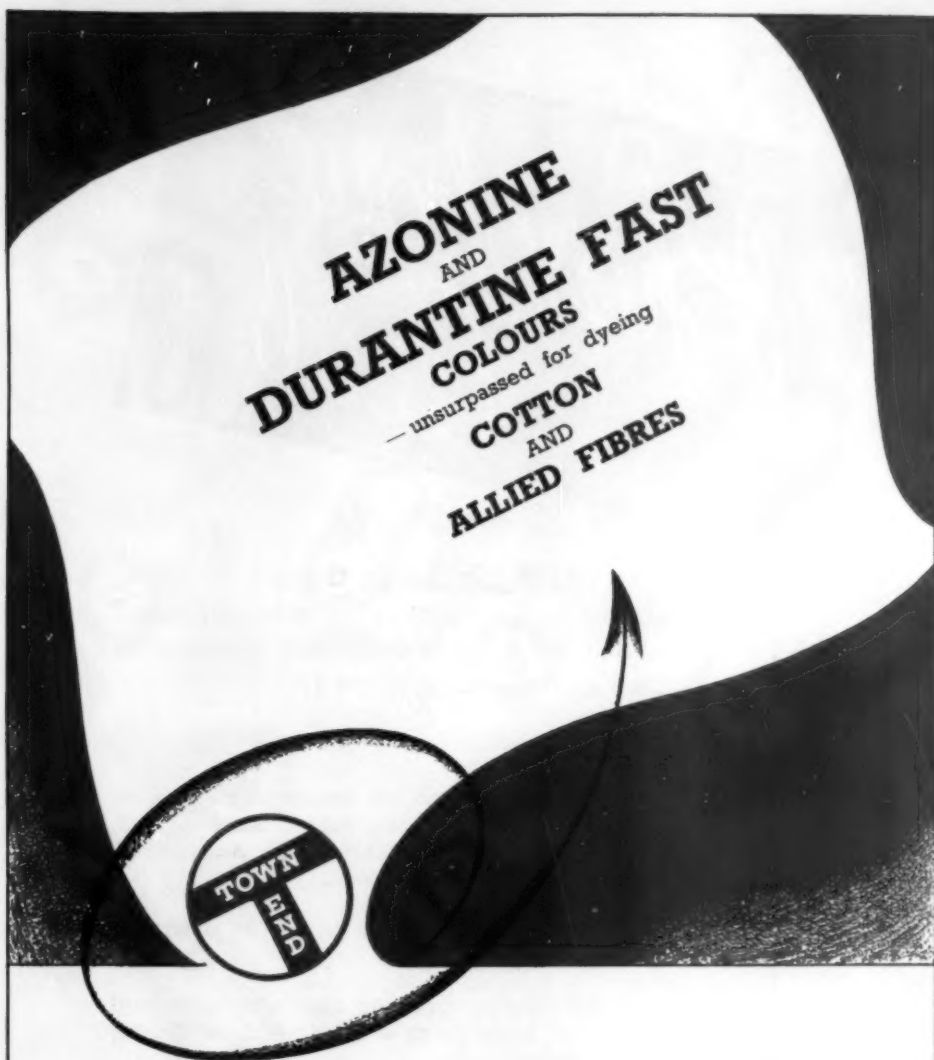
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70. ENDING

Uneven dyeing consisting in a continuous change in colour from one end of a length of fabric to the other, or a difference in colour between the bulk and the end of a length of fabric.

71. LEVELLING ACID DYE

An acid dye which migrates readily in the presence of dilute mineral acid.

72. GREY

(adjective)

Description of textile material before it has been scoured, bleached, or dyed.

73. KIER BOILING (KIERING)

Prolonged boiling of cellulosic materials with alkaline liquors in a large steel container (kier) at or above atmospheric pressure.

74. LEUCO DYE

A reduced form of a dye from which the original dye may be regenerated by oxidation.

75. LISTING

An uneven dyeing defect consisting in a variation of colour between the selvages and the middle of the dyed fabric.

76. METACHROME PROCESS

See 66. *Chromate Dyeing Process*

77. MILLING DYE

A dye which is fast to acid or alkaline milling treatments.

78. MOCK CAKE

A package of yarn produced by winding on to a collapsible mandril or former, which is removed after the package has been formed. Usually the package has the same dimensions as a rayon cake.

NOTE—It is usually built up from the inside to the outside, in contradistinction to the cake.

79. MOLTEN-METAL DYEING PROCESS

A method of dyeing in which fabric is impregnated with an aqueous solution of a dye and then passed through molten metal at a temperature not greater than 100°C.

80. BLOTCH

The uniformly printed background of a pattern.

81. NEUTRAL-DYEING ACID DYE

An acid dye which has useful substantivity for wool when applied from a neutral dyebath.

82. ONIUM DYE

A water-soluble cationic dye in which the ionisable group is an ammonium, sulphonium, phosphonium, or oxonium radical.

83. PAD-STEAM PROCESS

A process of continuous dyeing in which fabric in open width is padded with dye and any necessary assistants, and is then steamed.

84. PIGMENT PADDING

The application of vat dyes by padding with an aqueous dispersion of the unreduced vat dyes.

85. POTTING

A finishing process for wool cloths in which a roll of fabric is treated in boiling water for several hours to set the fabric and impart a firm and lustrous finish.

86. PREBOARDING

The operation of setting knitted garments or hose in a desired shape by the application of dry or moist heat to the article supported on a former.

87. RESIST

A substance applied to a textile material to prevent the uptake or fixation of a dye or finishing agent in a subsequent operation.

The substance may function either by forming a mechanical barrier, by combining chemically with the dye, or by altering conditions (e.g. pH value) locally so that development cannot occur.

88. RESIST (RESERVE) STYLE

A style of printing in which undyed material is printed with resists to give on subsequent dyeing or developing a white pattern on a coloured ground, or a pattern of contrasting colour to the ground by incorporating suitable dyes in the resist print paste.

89. RESTRAINING AGENT

A product which, when added to a dyebath, reduces the equilibrium exhaustion.

90. RETARDING AGENT

A product which, when added to a dyebath, reduces the rate of dyeing but does not affect the final exhaustion.

91. SETTING

The process of conferring stability of form on fibres, yarns, or fabrics, generally by means of hot water or moist or dry heat.

92. STRIPPING

Destroying or removing dye or finish from a fibre.

93. DEVELOPER

An azo coupling component used to couple with a diazotised dye or base on the fibre.

94. DISCHARGE PRINTING

A method of printing in which dyed fabric is printed with a chemical composition which destroys the dye locally to give a white pattern, and in which a second dye may be applied simultaneously with the discharge to produce a pattern of a different colour.

95. EFFECT THREADS

Threads, inserted into a fabric during manufacture, of such a character that they will present a different appearance from that of the bulk of the fabric in the final material.

96. MORDANT

A substance which is applied to a fibre to form with a dye a complex which is retained by the fibre more firmly than the dye itself.

97. MORDANT DYES

Dyes which are susceptible to fixation with mordants.

98. SCOURING

Freeing textiles from dirt and other extraneous matter by aqueous treatment with a detergent.

99. STOVING

Treating wetted wool, silk, or hair material in an atmosphere of sulphur dioxide in a chamber. (Wet stoving is treatment of a material with a strong solution of sulphur dioxide.)

100. SULPHUR DYE

A water-insoluble dye which is normally applied in the soluble reduced form from a sodium sulphide solution and subsequently reoxidised to the insoluble form on the fibre.

101. SWEALING

Undesirable migration of dye occurring between the dyeing and the drying of yarn or fabric.

102. VAT DYE

A water-insoluble dye, usually containing keto groups, which is normally applied to the fibre from an alkaline aqueous solution of the reduced enol form, the latter being subsequently reoxidised in the fibre to the insoluble keto form.

103. VAT

Noun—(a) A vessel in which dyeing is carried out.

(b) A dye liquor containing a reduced (leuco) vat dye together with the necessary alkali and reducing agent.

Verb—To bring a vat dye into solution by the combined action of alkali and reducing agent.

104. WINCH DYEING MACHINE (WINCH)

A dyeing machine in which an endless length of fabric is drawn through the dyebath by a reel or drum rotating above the surface of the dye liquor.

105. COUPLE

(verb)

To combine a suitable organic component, usually a phenol or an arylamine, with a diazonium salt in such a way as to form an azo dye.

AATCC President

Mr. Raymond W. Jacoby, of Mountain Lakes N.J., has been elected the thirteenth president of the AATCC.

Textile Institute Medal Awards

The Textile Institute Medal, awarded for distinguished services to the textile industry in general and to the Institute in particular, is to be presented to Mr. W. Crossley and to Dr. P. W. Cunliffe, who is Chairman of the Society's Fastness Tests Co-ordinating Committee.

Award of A.S.T.M. Medal to Dr. J. H. Dillon

Dr. John H. Dillon, Director of the Textile Research Institute, Princeton N.J., received on 17th March 1955 the Harold De Witt Smith Memorial Medal for 1955. This is the sixth award of the medal by Committee D-13 on Textile Materials of the American Society for Testing Materials. The medal is awarded at intervals of not less than one year by Committee D-13 for outstanding achievement in research on fibres and their utilisation.

International Federation of Associations of Textile Chemists and Colourists

A meeting of the Council of the Federation held in Paris on 23rd September 1954, at which were represented Belgium, France, Germany, Holland, Italy, and Switzerland, stood in memory of the late Professor R. Haller and Ing. Louis A. Driessen (President of the Nederlandse Vereniging voor Textielchemie).

An Italian invitation to hold the next international congress in Florence in September 1956 was accepted.

Verein der Textil-Chemiker und -Coloristen (V.T.C.C.)

The annual congress of the V.T.C.C. will be held at Baden-Baden on 4-7th May 1955.

Owing to pressure of other duties, Dr. Joachim Müller has resigned as secretary of the V.T.C.C., and the work has been taken over by Professor Helmut Zahn, z. Hd. Melland Textilberichte, Heidelberg, Friedrich-Ebert-Platz 3, Germany.

German Dyers' Congress

Wuppertal, 10-13th June 1955

This year the Deutscher Färbertag, the annual congress of German dyers, will be held in Wuppertal,

an old established centre of the textile industry which contains many works devoted to bleaching, dyeing, printing, and finishing. Information about the congress may be obtained from the Secretary of the Verein Deutscher Färber e.V., Dr. K. H. Möller-Klepzig, Düsseldorf-Oberkassel, Luegallee 68, Germany.

International Exhibition of Dry Cleaning and Dyeing

Munich, 15-24th July 1955

This will be held in the Ausstellungspark, and at the same time an international congress will be held. The exhibition is being organised by the Verein für Handwerks-Ausstellungen und Messen e.V. (Munich 12, Theresienhöhe 14, Germany) under the auspices of the Comité International de la Teinture et du Nettoyage (Paris) and the National Federation of Dyers and Dry Cleaners in Western Germany.

International Congress on the Documentation of Applied Chemistry

London, 23rd-25th November 1955

This will be held under the auspices of the International Union of Pure and Applied Chemistry. Enquiries may be made of the Honorary Secretary at 56 Victoria Street, London S.W.1.

The Polarographic Society

The inaugural meeting was held in London on 9th November 1954, the objects of the society being to promote the science of polarography and to provide educational facilities in polarography. Further information about the society can be obtained from V. S. Griffiths, Chemistry Department, Battersea Polytechnic, London S.W.11.

Centre for Research on Macromolecules, Strasbourg

This recently opened foundation of the French National Centre for Scientific Research consists of a physics section, an X-ray section concerned with the determination of the crystallinity of fibres, a biological section, and a chemical division comprising two sections concerned respectively with the synthesis of macromolecules of low molecular weight and well defined constitution and with the study of polymerisation reactions.

U.S.A. File of Coal-tar Products

The U.S. Bureau of Customs is to establish a central reference file of samples of coal-tar products of domestic manufacture, together with relevant technical and pattern-card data, to enable customs officers to ascertain the tariff status of imported coal-tar products, including dyes and intermediates.

Azoton—Cyanoethylated Cotton

The first pilot plant for the semi-large-scale investigation of this process has recently been installed at Rossville, Georgia, U.S.A. The Institute of Textile Technology of Charlottesville, Virginia, U.S.A., has coined the name *Azoton* for the new material.

Mark for All-silk Goods

The Silk Centre (49 Park Lane, London W.1) has introduced a distinguishing mark, embodying the word *silk*, for application to goods made entirely of silk, whether thrown or spun. It may not be applied to mixtures of silk with any other fibre.

Terital Polyester Fibre

The Società Montecatini has adopted the brand name *Terital* for the polyethylene terephthalate

fibre which it is to start to produce in Italy this year.

Dyeing and Printing in Burma

The Burmese Minister for Industries has opened the Co-operative Dyeing and Printing Works at Thamaing, near Rangoon. This is apparently the only works of this type in Burma.

"Plastics"

The Council of the British Plastics Federation has stressed that the adjective "plastic" should not be misused to indicate that articles are made from various types of plastics. In accordance with *B.S. 1755: Glossary of Terms used in the Plastics Industry*, the adjective should be *plastics*.

New Books and Publications

The Physical Chemistry of Dyeing

By Thomas Vickerstaff. 2nd edition 1954. Pp. viii + 514. London: published for Imperial Chemical Industries Ltd. by Oliver & Boyd. Price, 42s. 0d.

A new edition of Dr. Vickerstaff's book is welcome, both for itself and as some indication of the fact that its original must have been reaching the readers for whom it was intended on a satisfactory scale.

The reviewer has said elsewhere regarding the first edition, and it is worth saying again, that this book does something more than bring together all the important contributions which physical chemists have made to our understanding of the dyeing process. The author's own insight into the nature of the problems discussed has enabled him to produce a book which presents essentially the basis for a general theory of dyeing. It is now about twenty-five years since the English school of physicochemists—dyers began to produce papers which laid the foundations for much of our present understanding, and work by Neale, the Courtaulds school, the British Cotton Industry Research Association, Dr. Vickerstaff and his colleagues, and others is well integrated into the general picture together with much else which the author's association with the dyemaking industry has made accessible to him.

One criticism of the first edition was that it did not include advances in the theory of dye diffusion which had been made between the writing of the book and its publication three years later. This has been attended to in the new edition, and the work of Wilson, Crank, and others is incorporated in the sections dealing with dyeing kinetics. Two further chapters, additional to the original book, concern the dyeing of cellulosic fibres with vat dyes and the dyeing of synthetic fibres other than the

polyamides, which were the sole representatives of that class considered in the first edition.

In the first of these chapters the discussion is adequate and brings out the need for further study of the vat dyebath, particularly with respect to (a) dye-reduction potentials on cellulose and (b) the kinetics of dye absorption. The latter provides the most pressing need so far as concerns technical dyeing. Strike and levelling (migration) are the vital features in the bulk application of leuco vat dyes. To elucidate fully the relation of these dye characteristics to each other and to the overall dyeing process will require new and precise data on both dye affinity and rate of diffusion in fibres. The original data of Boulton and Morton are, according to Dr. Vickerstaff, still the most satisfactory alternative: but this reviewer agrees with him that much further work is called for. Moreover, continuous methods of dyeing, involving the vat dyes in padding and steaming processes, call, in themselves, for a re-assessment of existing data and a new approach to their practical application.

The advent of the acrylic and polyester fibres has provided something of a crisis in the development of dyeing techniques, and in his new chapter on Synthetic Fibres Dr. Vickerstaff brings out the inherent difficulty facing the dyer in handling virtually non-swellable substrates. He provides a useful summary of the lines of attack now being followed towards resolving this but, of necessity, leaves the reader with a feeling that the empirical approach which makes use of existing dyes and techniques is producing palliatives of a very restricted kind. Dr. Vickerstaff gives an encouraging picture of possibilities inherent in the cuprous ion method, suggesting that, where suitable sites for dye-adsorption are lacking, they may, in the future, be introduced by simple mordanting. As a general method this certainly

would be preferable to e.g. the promotion of fibre tumescence by "carriers" in the dyebath. But . . . first find your mordants! Practical dyers with experience in controlling the adsorption of cuprous salts from unstable reducing systems must hope devoutly that they are there to be found.

A stimulating feature of the book is that, in a number of contexts, the author draws attention to problems which remain unsolved for lack of data, the finding of which, in general, should not present undue difficulties to the experimenter. This, one hopes, will lead more of his readers to attempt research in some of those fields of applied physical chemistry of which Dr. Vickerstaff gives so well informed an overall account.

The book is very well produced, and it is encouraging to note that, although the price is still high, the second edition has one hundred more pages of text and many more diagrams without an attendant increase in price.

J. BOULTON

Technical Manual and Year Book of the American Association of Textile Chemists and Colorists

Vol. 30. (1954). Pp. 582. New York: Howes Publishing Co. Inc.

The first two and the last parts of this volume comprise the Association's year book, and well show its vigorous, ever increasing, and more widely spreading activity, but it is as a technical manual that the book has the greatest appeal outside the U.S.A., this being especially true of Part III, which contains the AATCC official test methods, as these alone make the book a necessity to most tintorial technologists. The test methods appearing for the first time are Rapid Control Tests for Fastness to Gas Fading, Fulling, and Determination of the Electrical Resistivity of Fabrics. In addition, revised tests for fastness to washing, light, and snag are given. The list of American-made dyes is now to be revised biennially, the first revised list to appear in 1955, but, though the present list is the same as that which appeared in the 1953 Year Book, opportunity has been taken to make some necessary corrections. The list of Textile Chemical Specialties has been completely revised in accordance with information supplied by the manufacturers and, giving as it does the uses and chemical nature of each product listed, is a very useful list to have handy for reference. This book is indeed a work which should be readily available to all concerned with the colouring or finishing of textiles, and of which the Association may justly be proud.

C. O. CLARK

Crown Point Dye Works East Street, Leeds

The Story of a Family Business

Expanded and revised by H. Waddington.
Pp. i + 108. Leeds: Privately published 1953.
[Mimeographed.]

This book, which is an expansion of a previously published brochure, describes the history of over a century and a half of a family business which combined the dyeing of wool pieces with that of

dyeware grinders and dyewood cutters with customers widely spread among dyers in the West Riding. It is probably unique in its description of a dyehouse and its workings in the period embracing the changeover from manually powered winch dyeing machines (each took three men to operate it) heated by direct fire to machines both driven and heated by steam, from entire reliance on natural dyes to almost complete use of coal-tar dyes, of a dyehouse in idyllic rural surroundings and their gradual change until it was buried in the intestines of a great commercial and industrial city. It will undoubtedly become a standard book, to which all historians of dyeing will go and for which they will owe an eternal debt of gratitude to Mr. Waddington. But Mr. Waddington has put them further into his debt, for he and his nephews have ensured preservation of the records, pattern and recipe books of the firm, covering the period 1816-1947, by presenting them to Leeds University, where they are now available for use by all research workers into the history of the dyeing industry. As the book deals not only with the business but with the characters and social life of those connected with it, partners and workmen alike, as well as with the growth of Leeds from a pleasant country market town to the great industrial city it now is, it will also be invaluable to the social and the local historian.

Not all Mr. Waddington's opinions are beyond challenge—he is not always consistent in his comparisons of the fastnesses of natural and coal-tar dyes—but the only incorrect statements of fact noticed are that on p. 10, where he says that Perkin discovered aniline, "which was the first synthetic dye ever produced", when in fact what Perkin did was to make from aniline the first dye produced from coal tar; and a later statement that logwood is now used only on a small scale for tinting.

These errors in no way detract from the great merits of the book, which is a wonderful achievement for a man of Mr. Waddington's years—he was born in 1865. There is no doubt that in writing it he has produced what will come to be recognised as a classic of its kind, which will provide an imperishable memorial to the lifework of his grandfather, his father, and himself. It is to be hoped that his example will spur others on to a similar effort, for there is a great dearth of such books, and the number of people who can, from their own experience, write of the great change in dyeing practice that occurred in the last hundred years is dwindling rapidly: they ought to regard it as a duty to posterity to put their experiences on record in the way that Mr. Waddington has done.

C. O. CLARK

Handbook of Textile Fibers

Edited by Milton Harris. Pp. xii + 356. Washington, D.C.: Harris Research Laboratories Inc. 1954. Price, \$12.50.

For a long time there has been a need for a comprehensive and authoritative collection of data on textiles. The present work is the first serious attempt to provide this in the literature.

The preface says—

This Handbook is the outgrowth of an organized program started ten years ago by our staff to compile and edit data which are of special importance to the research worker in the field of textiles.

It is an ambitious work and covers a great range of topics. It starts with thirty pages of textile terms and definitions, which are neither co-extensive nor congruent with the Textile Institute definitions. Names, sources, constitutions, and microscopical appearances of fibres are given in an orthodox way for some thirty pages. There is a brief section on X-ray diffraction characteristics.

Then for about half the book there are tables of numerical data on the physical, chemical, and biochemical properties of fibres. Previously collections of data have been published by Herzog and others, but the present collection far exceeds any earlier one in range and detail. Nevertheless, it is not exhaustive. This is unquestionably the most important and original part of the book: any success the handbook may have will depend on the accuracy and comprehensiveness of these data. The information in the rest of the book is less important. It will be found either as fully or more fully in other publications. For example, the tests on the identification of fibres are reprinted without modification from the Textile Institute, because "this collection of tests is so thorough and well arranged".

Next are sections on yarn numbering and cost systems, useful miscellaneous chemical and physical data, and a summary of economic data on production and prices of textiles. These will probably be found useful by those without many reference books, but are sketchy compared with the range of data in the standard reference books such as the *Handbook of Chemistry and Physics* and the H.M.S.O. and F.A.O. surveys of fibre production data. Easy reference to the data is made possible by a full subject index.

A large reference collection of data can be tested only by examining it in places where what should be there is known. When this was done, quite a number of defects were noted. Despite this, the work will be found very useful as a guide to what has been published. J. M. PRESTON

Die neuzeitlichen Textilveredlungs-Verfahren der Kunstfasern

1. Ergänzungsband—Die Patentliteratur und das Schrifttum von 1950–1953

By F. Weber and A. Martina. Pp. xv + 381. Vienna: Springer-Verlag. 1954. Price, DM 58.00.

The original volume by Weber and Martina, published in parts during 1950–1951 (J.S.D.C., 67, 152, 451 (1951); 68, 24, 69, 214 (1952)), quickly established itself as a standard source book for the literature, more especially the patent literature, on the finishing of man-made fibres. The need for a supplementary volume to bring the information up to date has been felt for some time, and all users of this compilation will welcome this volume, which covers the 1950–1953 literature. It contains references to over 5,000 patents and more than

2,000 references to other literature. This is some measure not only of the need for and usefulness of this volume but also of the vast amount of work now being done in this field all over the world. The compilation still contains unexpected sections, e.g. those on carotting and chlorination of wool, and it is hard to justify their inclusion. It is difficult to see of what use the dye index can be; how much more useful would an author index be! It is to be hoped that Weber and Martina will continue their good work, so that we can look forward to regular issues of supplementary volumes to keep the compilation up to date. At the same time it is to be hoped that they will publish an author index as speedily as possible, and not wait till their growing collection of references justifies the publication of the second supplementary volume. C. O. CLARK

Synthetische Wasch- und Reinigungsmittel Chemie, Klassifikation, Technologie, Komposition, Anwendung, Untersuchungsmethoden und wirtschaftliche Bedeutung

By H. Stüpel. Pp. 568. Stuttgart: Konradin-Verlag Robert Kohlhammer. 1954. Price, DM 68.00.

The rapid development of the synthetic detergent industry since about 1930 has created the need for a practical textbook on the subject. Dr. Stüpel's book on "Synthetic Detergents and Washing Agents" can be regarded as a most useful and thorough contribution to the literature. The book by J. W. McCutcheon on "Synthetic Detergents" published in 1950 (New York: MacNair-Dorland Co.) was less comprehensive. Although surface-active agents and synthetic detergents were first used by the textile industry, it has now been established that 90% of the detergent production is absorbed by the household detergent manufacturers, and the balance by the textile industry.

The author presents and discusses the general aspects of synthetic detergents in a very concrete and practical form; as chief chemist of a Swiss soap factory, he increases the value of his book by commenting critically on the publications of other workers and by reporting the results of his own practical experiments and work.

Chapter I reviews the fundamentals of detergency, the correlation between washing effect and the chemical structure of detergents, and the relation between surface activity and such characteristics as emulsification, foaming, wetting, dispersion, and detergency.

Chapter II describes the chemical structure of anionic, non-ionic, and cationic surface-active and "wash-active" substances, although cationic compounds do not possess any detergent effect. It deals at length with sulphonated oils and fats, sulphated and sulphonated amides and amines, fatty alcohol sulphates, alkylarylsulphonates, alkylsulphonates, secondary alkyl sulphates, and related products. The manufacturing methods for these products are described in great detail. There is an alphabetical list of the names of about 150 German, French, American, Swiss, and (a few)

British products, their chemical constitution, content of wash-active substance (in percentage), and group. Also, comparative tables are given of wash and surface activity, foaming power, lime-soap dispersion, and other characteristics of the various groups of synthetic detergents.

The author discusses in Chapter III the compounding of synthetic detergents by the combination of various groups of wash-active substances, by combining synthetic detergents with soap, by adding inorganic electrolytes, such as silicates and phosphates, and such organic substances as carboxymethylcellulose and optical brightening agents. The effect of the addition of such builders on the performance of synthetic detergents is of the greatest importance and of fundamental interest. In this chapter Dr. Stüpel gives the chemical analysis of some of the well known household detergents marketed in America, Germany, Switzerland, and Britain.

Chapter IV describes the manufacturing methods for liquid, paste, and solid detergents, with a special section on the modern technique of spray-drying; while Chapter V outlines the application of "wash-active" substances in industry and household.

Chapter VI is a summary of the properties and characteristics of synthetic detergents, their soil-suspending power, their dermatological and biological action. It discusses also the corrosive action of synthetic detergents, which is, incidentally, more pronounced than that of soap, and which can be caused also by the builders present. Chapter VII gives analytical and evaluation testing methods.

Chapter VIII summarises the economic importance and development of the detergent industry in various countries. It is gratifying to observe from Table 204 that Britain belongs to the countries of high cleaning standard and ranks third, with Belgium first and the U.S.A. second, in the *per capita* consumption of soap; Brazil, Japan, Java, Panama, and India are at the bottom of the list. There are also tables showing the consumption of soap and synthetic detergents in the U.S.A., Britain, and other countries. On the question of the controversy between soap and synthetic detergents in industrial and household use, the author predicts a "peaceful coexistence" (to use a phrase of which we hear so much in other spheres of life), with an increased consumption of synthetic detergents, in years to come.

As can be seen, Dr. Stüpel presents a very interesting and extensive study: the table of contents occupies fifteen pages; there are excellent diagrams, tables, and graphs, a study of which would be of interest to the reader not conversant with German. There are over 2000 literature references; all are assembled at the end of the volume instead of at the end of each chapter. There are subject and author indexes. A comprehensive bibliography is included, to which should be added the book (since published) by J. C. Harris *Detergency Evaluation and Testing* (reviewed in J.S.D.C., 70, 451 (Oct. 1954)).

The book by Dr. Stüpel is pleasantly produced and printed, and the author and the publisher may be congratulated on a book of such importance and interest: it should be translated into English.

A. F. KERTESS

Colorimetric Methods of Analysis
Including Some Turbidimetric and Nephelometric Methods

Volume III
Organic—I

By F. D. Snell and C. T. Snell. 3rd edition 1953. Pp. viii + 606. New York: D. Van Nostrand Co. Inc. Price, 93s. 6d.

In these days new analytical procedures and instruments proliferate like enthusiastic amoebae, and many classical tools of gravimetry and titrimetry are saved from joining the alchemist's alembic only by sheer, proved usefulness. Colorimetry proceeds, however, from strength to strength, and if Beer is not always best, he is at least an increasingly useful fellow. The scope and the magnitude of colorimetry are clearly demonstrated by the very existence and nature of the fine series of books of which the present volume is a part. More than six hundred pages are devoted to the determination of a score of broad classes of organic compounds by straightforward absorptometric, nephelometric, and turbidimetric methods, supplemented in some instances by chromatography—an aspect of colorimetry which has already outgrown its name, and which is almost beyond confinement to a single volume of reasonable size.

This is a strictly practical book, well suited to the analyst who has something on his bench, and merely wants to know how best to determine it. Structural formulae are conspicuous by their absence, line formulae are few and far between, and theoretical considerations are brief, simple, and directly helpful. Whilst the numerous procedures detailed are clearly the first choices of the authors, so many others are included in general discussion that literature references run into the hundreds: the book is thus valuable both as a practical handbook and as a survey. In many cases the determination of a single substance in a variety of substrates is described.

The reviewer cannot possibly know the extent of the branching interests of his readers, but it is easy to compile a list (which could be expanded to formidable proportions) of compounds whose determination is discussed, and which are clearly of interest to J.S.D.C. readers—phenols, naphthols, and quinones (substituted and unsubstituted); polyvinyl alcohol; glycerol; gossypol; pentoses, hexoses, heptoses, polysaccharides, and carbohydrates (including cotton cellulose, hemicellulose, methylcellulose, dextran, and starch); lanolin; ethylenediaminetetra-acetic acid; tannins; alginic acid; cystine and cysteine sulphur; and many chlorinated compounds such as DDT and DMC.

The book is stoutly and attractively bound, it is clearly printed on good paper, and the illustrations

and diagrams are clear and valuable. There are a few printing errors but these, like the "h" missing from "pyrethrins" under Fig. 30 (p. 466), are easy to spot, and should trouble no intelligent reader. This book should run to many editions.

J. W. DUARTE

**Methoden der Organischen Chemie
(Houben-Weyl)**

Band IV

Allgemeine Chemische Methoden

Teil 2

Edited by Eugen Müller. 4th edition 1955. Pp. xxviii + 1004. Stuttgart: George Thieme Verlag. Price, DM 152.00.

This collection of general chemical methods employed throughout organic chemistry contains all the most important material published in the literature and in patents up to the end of 1953, and in many cases up to the middle of 1954. The contents are dealt with in eleven chapters as below.

1. *Acid-Base Catalysis*—The subject matter is prefaced by a short theoretical discussion of the various reaction mechanisms proposed by Brönsted, Lowry, Lewis, etc., followed by sections on the activation of carbon-oxygen bonds and carbon-carbon double and triple bonds, on decarboxylation, and on nitrogen-containing compounds including the decomposition of aliphatic diazo compounds, the Wolff-Kishner reduction, and the various rearrangements such as the *N*-halogeno-*N*-acylanilide, *NN*-diacylanilide, Fischer-Hepp, arylnitramine, arylhydroxylamine, benzidine, and diazoamino-aminoazobenzene transformations. The chapter concludes with a note on practical considerations.

2. *Catalysis by Complex Cations and Anions*—Here general observations are made on molecular activation by formation of complex compounds, and are followed by special reference to activations of individual linkages.

3. *Methods for the Preparation of Catalysts and Mixed Catalysts*—These are subdivided for general and special requirements.

4. *Procedure for Heterogeneous Catalytic Reactions*—A general section, dealing with processes and conditions, precedes the consideration of apparatus and of special catalytic reactions for isomerisation, hydrogenation with molecular hydrogen, dehydrogenation with elimination of

molecular hydrogen, and catalytic oxidations with molecular oxygen. Following comes a special section which concerns reactions at olefin linkages without formation or rupture of a C-C bond, the formation of a C-C bond by addition reactions at olefin double bonds, addition reactions of hydroxyl compounds, ethers, acetals, and at a triple bond, aldol condensations, and cleavage reactions with and without rupture of a C-C bond.

5. *General Methods for Pyrochemical Reactions*—These include reactions promoted by internal electrically heated metallic wires and rods, by external heating through special installations and also in fused-metal baths (e.g. of lead), and devices for the pyrolysis of micro amounts of material.

6. *Electrochemical Reactions*—In this section, theories of mechanism are first discussed and then special processes at the anode and the cathode are described.

7. *Methods for the Preparation of Optically Active from Inactive Compounds*—These mainly concern the resolution of racemates and asymmetric syntheses.

8. *Preparation of Organic Compounds with Labelled Elements*—These include compounds containing ^{14}C , deuterium and tritium, ^{15}N , ^{18}O , ^{35}S , ^{82}Br , ^{131}I , and ^{32}P .

9. *Methods for the Preparation and Transformation of Large Ring Systems*—General conditions precede special processes, which include depolymerisation, ring extension, ring closure, ring contraction, and transformability of many-membered rings.

10. *General Methods used in Biochemical Reactions*—These concern ferments and micro- and macro-organisms.

11. *Methods used for Microbiological-Chemical Reactions*—These include the use of cultures, nutrients, inoculants, and temperature.

The range of this volume may be appreciated from the fact that the author index occupies 44 pages of names in three columns and the subject index 58 double-column pages.

The favourable opinion already recorded with respect to Volume VIII (cf. *J.S.D.C.*, **69**, 133 (1953)) applies with equal force to the present volume, which will be welcomed by all organic chemists.

H. H. HODGSON

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., 68, 23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index

I—PLANT; MACHINERY; BUILDINGS

PATENTS

Continuous Wet Treatment of a Lap of Staple Length Man-made Fibres. American Viscose Corpn.

USP 2,665,189

A method of rapidly wet processing a lap of staple fibres using machinery which occupies little space. C. O. C.

Applying Liquid to Sheets or Webs. United States Atomic Energy Commission.

USP 2,665,660

Liquid is directly applied by passing the web through the slit between two non-absorbent closely fitting elements, the liquid being applied at or near the closely adjacent parts of the elements. Two components of the liquid may be mixed together at the point of application. C. O. C.

Continuous Heat-treatment of Fabrics of Glass or other Man-made Fibres. Alexander H. Kerr & Co.

USP 2,665,125

A furnace for the continuous removal of size from glass fibre fabrics followed by application of silicones, unsaturated chromium complexes, etc. C. O. C.

Applying a Reduced Pressure to a Web of Material. E. A. R. Mathins.

BP 720,296

Apparatus for applying a reduced pressure to one side of a web of material comprises a top frame of a suction-box, and a series of rollers (½ in. to 2 in. in diameter) disposed across the upper part of the frame in parallel lines transverse to the direction of movement of the web, the rollers having end spindles of smaller diameter and being separate from those of other rollers to allow of independent rotation. At least three rollers are provided in each transverse line, the upper generatrices of the rollers are in the same horizontal plane as the sides of the box frame, a plurality of bearings, of fibre-filled resinous composition, is provided for the spindles and means for lubricating the bearings are provided. The rollers in one row may be staggered relative to those in other rows, to bridge the gaps at the bearings and the end rollers are adjustable axially for different widths of webs. The box frame may carry resilient side sealing strips parallel with the direction of the wire gauze belt and on which the belt seats. S. V. S.

Stencil Printing Apparatus. Velverny Corpn.

USP 2,665,634

A method of combined roller and flock printing in which registry is assured of the two sets of prints. The main drive shaft is driven by a chain which in turn has a supplemental shaft driving a stencil roller as well as a main gear driving several intaglio rollers. The drum which carries the fabric is driven by contact with the stencil roller as well as the intaglio rollers. C. O. C.

Preboarding Nylon Hose. L. Heldmaier.

BP 719,795

Nylon hose are continuously preboarded or plasticised by placing on metal forms carried by an endless conveyor through a chamber containing radiant heaters and in which hot air is circulated in closed circuit. Thermostatic control of the air stream is provided and the machine is particularly designed to economise in floor space. G. E. K.

Alphabetical List of New Products (Colouring Materials, Chemicals, and Equipment) November 1953–November 1954 (IV this page).

Applying Transfers to Ceramics (IX p. 162).

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

PATENTS

Size. Shell Development Co.

USP 2,665,983

A concentrated size readily dispersed in water consists of a mixture of hydrocarbons and a polyphosphoric acid compound preferably together with a mutual solvent. The composition is especially suitable for paper. Emulsions produced from them are capable of phase inversion. C. O. C.

Size. American Viscose Corpn.

USP 2,666,038

A composition consisting of an aqueous dispersion of a predominantly naphthenic mineral oil containing a water-soluble or dispersible binding colloid and, as emulsifier or stabiliser, a water-soluble or dispersible solid ester of m.p. < 30°C. of a fatty acid of > 11C with a polyhydric or anhydro polyhydric alcohol, has many uses as a textile size, e.g. as a conditioning agent to prepare a textile to receive a coating of rubber latex. C. O. C.

Phosphorus Oxychloride-Ammonia Reaction Products as Flame-retarding Agents. Monsanto.

USP 2,661,263

A flame- and glow-retardant finish is imparted by application of a mixture of antimony and titanium compounds together with the water-soluble, alkaline condensate obtained by reacting phosphorus oxychloride with anhydrous ammonia.

USP 2,661,311

The above condensate alone is used.

USP 2,661,341

Incorporation of 3–90% of the above condensate into a resinous phenolic body imparts a flame-retarding finish.

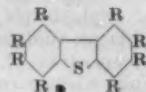
USP 2,661,342

The above condensate and an aminoplast resin yield a permanent fireproof with no loss in strength. C. O. C.

Dibenzothiophenes as Fungicides. Phillips Petroleum Co.

USP 2,665,234

Compounds of formula—



(R = same or different, H, Alk or alkenyl of < 4C, Hal, nitro, cyano, amino or carboxy) are effective fungicides for use with textiles, leather, etc. C. O. C.

Modified Melamine-Formaldehyde Resinous Condensates for imparting High Wet Strength to Paper and as Textile Finishes. Monsanto.

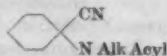
BP 720,068

A water-soluble melamine-formaldehyde resinous condensate is produced by heating a solution of melamine, formaldehyde and salicylic acid or an alkali metal salt thereof, the acid or salicylate being present to the extent of 0.5 to 2 (or 0.8 to 1) molecule per molecule of melamine. The reactants are mixed with water, heated gently until a clear solution has formed, and then further heated until a product of the desired molecular weight has formed. They are used for imparting high wet strength to paper and as textile finishes. S. V. S.

Insect Repellents. Cilag.

BP 721,081

Compounds of formula—



(Acyl = an aliphatic, araliphatic or aromatic acyl radical), e.g. cyclohexyl-1-cyano-1-(N-propionyl-N-methyl)-amine, are highly efficient insect repellents. C. O. C.

Alphabetical List of New Products (Colouring Materials, Chemicals, and Equipment) (IV below).

Delayed-action Catalysts for Use in Formaldehyde Resin Finishes (X p. 163).

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Alphabetical List of New Products (Colouring Materials, Chemicals, and Equipment) November 1953–November 1954. Anon. *Amer. Dyestuff Rep.*, 43, 805–836 (6 Dec. 1954).

Formation of *o*-Xylene through 4:5-Dimethylene-cyclohexene. J. E. Ladbury and E. E. Turner. *J.C.S.*, 3885–3886 (Nov. 1954).

Infrared Spectra of the Monohalogenonaphthalenes.

J. Ferguson and R. L. Werner. *J.C.S.*, 3645-3647 (Nov. 1954).

Preparation of Alkylanilines. W. Gerrard and G. J. Isaaccke. *Chem. and Ind.*, 1538 (11 Dec. 1954).

The anilino compound $(RO)_2P(O):NH-C_6H_5$, prepared from the dialkyl phosphorochloridate $(RO)_2P(O)Cl$, when heated at 240–250°C. gives the corresponding alkylaniline, C_6H_5-NH-R , where R = alkyl group. When R = C_2H_5 , $n-C_3H_7$, and $n-C_4H_9$, the yields of the alkylanilines are 95, 94, and 95% respectively.

H. H. H.

Oxidations with Lead Tetra-acetate. II—Oxidation of Primary Aromatic Amines. K. H. Pausacker and J. G. Soroggio. *J.C.S.*, 4003-4006 (Nov. 1954).

The oxidation is described of some monosubstituted anilines and of α - and β -naphthylamine by lead tetra-acetate in acetic acid soln. In most cases, the corresponding azo compound is formed, either alone or with other products. The % yields are shown in parentheses, the second and third figures indicating the yields when phenyl iodosoacetate is used in benzene and in acetic acid, respectively—*aniline* (5, 95, 0), *o*-toluidine (23, 42, 0), *m*-toluidine (18, 56, 0), *p*-toluidine (4, 6, 0), *o*-chloroaniline (56, 39, 25), *m*-chloroaniline (39, 60, 34), *p*-chloroaniline (35, 55, 24), *o*-anisidine (10, 3, 1), *p*-anisidine (16, 5, 4), *o*-nitroaniline (15, 0, 71), *m*-nitroaniline (40, 65, 61), *p*-nitroaniline (43, 53, 63), α -naphthylamine (0, 3, 0), β -naphthylamine (0, 0, 0), and *p*-phenetidine (26, 7, -).

H. H. H.

Sensitisation of the Photoconductivity of Silver Iodide by Organic Dyes. A. G. Goldsman and I. A. Akimov. *Zhur. Fiz. Khim.*, 27, 355-361 (1953); *Chem. Abs.*, 48, 13497 (25 Nov. 1954).

The photoconductivity of AgI films treated with dyes was determined as a function of wavelength λ in the 400–750 m μ . range. The dyes and λ in m μ . for maximum photoconductivity for each are—Methyl Green 620, Crystal Violet 480–540, Fuchsin base 500, Methylene Blue 580, Iodococin 520, Erythrosin 540, Safranin 480, Fluorescein 480, Bismarck Brown 546, Congo Red 540, Neutral Red 480–540, Eosin 520, Methylene Green 626, Na eosin 520, Methyl Violet 480–540, Bromophenol Blue 600, dimethylaminoazobenzene 480–540.

C. O. C.

Refined Free-electron Model of Porphin and Tetrahydroporphin. T. Nakajima and H. Kon. *Science Rept. Tohoku Univ., Ser. I*, 37, 164-170 (1953).**Colours of Triphenylmethane and Acridine Dyes.** H. Kon. *Ibid.*, 171-174; *Chem. Abs.*, 48, 13404 (25 Nov. 1954).

A refined free-electron model is used for study of the electronic structures and spectra of triphenylmethane and acridine dyes. Wave functions and secular equations, based on this model, are set up for both classes of dyes, and numerical values of energy levels are calculated from these. Comparison of the calculated first absorption bands and the observed centres of gravity of the singlet-singlet and singlet-triplet transitions for *p*-rosaniline, Crystal Violet, dimethylamino-*p*-rosaniline, Acridine Orange, and proflavine shows the method incapable of quantitatively explaining spectral shifts caused by change in the structure of a dye, but able to predict satisfactorily the general trend produced by such changes; e.g. the more electronegative the end group, the shorter the wavelength of an absorption band. This is clearly shown by inspection of the charge densities at the positions subjected to structural change.

C. O. C.

Derivatives of the Coloured Compounds formed by Condensation of Furfuraldehyde with Aromatic Amines. Compounds of Furfuraldehyde with Two Molecules of Aromatic Amines. J. C. McGowan. *J.C.S.*, 4032-4034 (Nov. 1954).

The (probable) 2-anilino-1:2:3:4-tetrahydro-3-oxo-1-phenyl- and 1:2:3:4-tetrahydro-3-oxo-2-*p*-toluidino-1-*p*-tolyl-pyridines previously reported by McGowan (*ibid.*, 777 (1949)) as resulting from the treatment of the corresponding Stenhouse dyes with aq. NaOH are now more conveniently prepared by boiling together aniline and *p*-toluidine respectively with furfuraldehyde in methanol.

H. H. H.

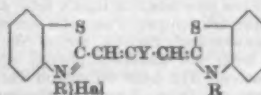
Some Tautomeric Acridines. R. M. Acheson, M. L. Burstall, C. W. Jefford, and B. F. Sansom. *J.C.S.*, 3742-3746 (Nov. 1954).

Contrary to the conclusions reached by other workers using other methods, ultraviolet absorption measurements on solutions of 5-dimethylamino-, 5-imino-10-methyl-5:10-dihydro-, and 5-amino-acridine indicate that the last named compound is largely present in the tautomeric form of 5-iminoacridan under the conditions employed. Similarly, acridone and thioacridone are present in solution in the CO and CS form respectively.

H. H. H.

Syntheses of Cyanine Dyes. V—Syntheses of Antibacterial Thiocarbocyanines. Y. Mizuno, K. Shibata, E. Hase, T. Uemura, and M. Katayanagi. *J. Pharm. Soc. Japan*, 68, 282-284 (1948); *Chem. Abs.*, 48, 13684 (25 Nov. 1954).

Three methods are described for making 7 dyes of formula—



(Y = H, Alk, or aralkyl; R = Alk).

C. O. C.

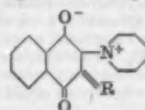
Trinuclear Sensitising Dyes—III. Y. Hishiki. *Rept. Sci. Research Inst. (Japan)*, 29, 526-532 (1953); *Chem. Abs.*, 48, 14213 (25 Nov. 1954).

Using $Cl-CH_2-COOH$ (I) or $Cl-CH(COOH)_2$ (II) instead of acetone as condensing agent in the usual synthesis of trinuclear dyes yields the indolocyanines, i.e. [bis-2-(1:3:3-trimethylindole)] [*y*-2'-(1':3':3'-trimethylindole)] pentamethylenecyanine-1₂ and -1₃, respectively. II is decarboxylated during the reaction to active I and so is more effective. The dyes are similar to the usual neocyanines.

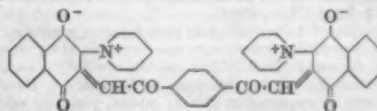
C. O. C.

Pyridinium Betaines from 2:3-Dichloro-1:4-naphthaquinone—New Type of Vat Dyes. B. Suryanarayana and B. D. Tilak. *Current Sci. (India)*, 22, 171-173 (1953); *Chem. Abs.*, 48, 14212 (25 Nov. 1954).

The products obtained by condensing 2:3-dichloro-1:4-naphthaquinone (I) with ethyl acetoacetate (II) and acetoacetylides in pyridine are deeply coloured pyridinium betaines, some of which are useful vat dyes. Condensing I and II gave the orange—



(R = $CH-COOC_2H_5$). Condensing I with $CH_2-CO-CH_2-CO-NH-C_6H_5$ in pyridine yielded a violet product of the same formula but R = $CH-CO-NH-C_6H_5$, and a red product where R = $CH-CO-CH_3$. These two products are pale pink vat dyes. Condensing I with Naphtol AS-LG and Naphtol AS-L3G in pyridine resulted in the red—

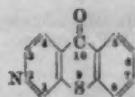


which yields reddish orange from a bluish-violet vat.

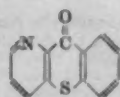
C. O. C.

Xanthenes and Thioxanthenes. V—Preparation and Properties of 9-Thia-2-aza-anthrone and 9-Thia-4-aza-anthrone. S. Kruger and F. G. Mann. *J.C.S.*, 3905-3910 (Nov. 1954).

3-Aminoisonicotinic acid condenses with thiophenol in alkaline soln. to form 4-carboxy-3-pyridyl phenyl sulphide, which $SOCl_2$ converts into the carbonyl chloride, and the latter, when heated with $AlCl_3$ in nitrobenzene, cyclises to 9-thia-2-aza-anthrone (I). 3-Aminopicolinic acid is similarly converted into 2-carboxy-3-pyridyl phenyl sulphide, and thence into 9-thia-4-aza-anthrone (II). The route is not applicable to the 9-oxa analogues.



(I)



(II)

H. H. H.

Anthraquinonoid Acid and Vat Dyes. K. Murata and K. Harada. *Bull. Fac. Engr., Hiroshima Univ.*, 3, 135-139 (1954); *Chem. Abs.*, 48, 10347 (10 Sept. 1954).

The colours of the powders and aq. soln. and the silk-dyeing properties are compared of a series of compounds derived by condensing 1-amino-4-bromo-2-sulphoanthraquinone with the two naphthylamines and various of their sulpho and hydroxy-sulpho derivatives. The hues on silk are violet to black except the compounds containing OH in the naphthalene nucleus, which are brown.

The colours of the powders and H_2SO_4 soln. and the hues obtained on cotton are compared also of a series of vat dyes obtained by condensing various amino derivatives of anthraquinone with 4:4'-dichlorodiphenyl and its 3:3'-dimethyl derivative. The hues on cotton vary; thus the compound obtained from 2-aminoanthraquinone and 4:4'-dichlorodiphenyl dyed pink, while that obtained from 1-amino-2-methylantraquinone dyed purple. R. K. F.

Preparation of Polycyclic Aromatic Hydrocarbons from Arylpropionic Acids. A. D. Campbell. *J.C.S.*, 3659-3669 (Nov. 1954).

2:3-Benzopyrene, naphtho(2':1:2)pyrene, 1:2:3:4:5:5a:6:11:11a:12-tetrabenzonaphthacene, and 1:2-benzopyrene are synthesised by decarboxylation and cyclo-dehydrogenation of the products of dimerisation of β -1-naphthyl-, β -2-naphthyl-, and β -9-phenanthryl-propionic acid, and the reaction product of β -phenylpropionyl chloride with β -2-naphthylpropionic acid, respectively. Several fluorenones are obtained by decarboxylation of the acids resulting from intramolecular cyclisation of the dimerisation products. H. H. H.

Synthesis of Pyrene. N. N. Saha and P. Bagchi. *Chem. and Ind.*, 1537-1538 (11 Dec. 1954).

2-Ethoxycarbonyl-2- β -phenylethylcyclohexanone when refluxed with a catalytic amount of $NaOC_2H_5$ in C_6H_5OH affords ethyl α -(β -phenylethyl)pyrrolate, cyclised (Dieckmann) by powdered Na in benzene under N_2 to sodium 2-oxo-3-(β -phenylethyl)-1-carboxylate, which, when refluxed with ethyl bromoacetate, gives ethyl 2-oxo-3-(β -phenylethyl)-cyclohexane-1-carboxylate-1-acetate. The latter is hydrolysed in glacial acetic acid-48% HBr to give a product $C_{21}H_{20}O_3$, which cyclises in $P_2O_5-H_3PO_4$ to hexahydropyrenol, and dehydrogenates with 30% Pd-C at 260°C. to pyrene. The synthesis could easily be extended to 3:4-benzopyrene. H. H. H.

Fluorescence Spectra of Coronene and 1:12-Benzoperylene at Low Temperatures. E. J. Bowen and B. Brocklehurst. *J.C.S.*, 3875-3878 (Nov. 1954).

Indoles. I—Formylation of Indole and Some Reactions of 3-Formylindole. C. F. Smith. *J.C.S.*, 3842-3846 (Nov. 1954).

The yields of 3-formylindole and 2-formylpyrrole, when indole and pyrrole respectively are formylated by means of $POCl_3$ in excess of dimethylformamide, are 95.5% and 83%. The mechanism of this reaction is discussed. Urorosein perchlorate is prepared in almost quant. yield by addition of perchloric acid to an equimol. mixture of 3-formylindole and indole in methanol. H. H. H.

Aromatic Keto-enols. III—Some Heterocyclic Quinols. A. Blackball and R. H. Thomson. *J.C.S.*, 3916-3919 (Nov. 1954).

Alkylated 4:7-dihydroxyindoles (I) are prepared by the Biechler synthesis; e.g. condensation of 2:5-dimethoxy-aniline with 1-bromoethyl methyl ketone gives 4:7-dimethoxy-2:3-dimethylindole, from which the quinol (I: $R = CH_3$) is obtained by demethylation with $AlCl_3$. Attempts to tautomerise 5:8-dihydroxyquinoline, 4:7-dihydroxythionaphthen, 4:7-dihydroxy-2:3-dimethylindole, and 1:3- and 2:3-dihydroxynaphthalene were unsuccessful.

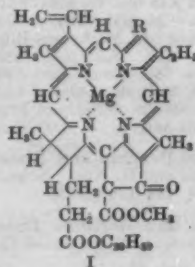
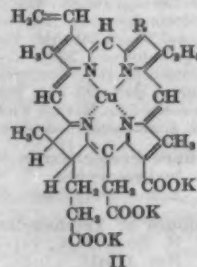


(I)

H. H. H.

Chlorophyll by Solvent Extraction. M. A. Judah, E. M. Burdick, and R. G. Carroll. *Ind. Eng. Chem.*, 46, 2262-2271 (Nov. 1954).

A commercial process for the production of chlorophyll (I) is described in detail. The process is based on the extraction of dehydrated alfalfa leaf meal (containing approx. 0.4% I) with 80-90% hexane-acetone. The extract contains carotene, xanthophyll, plant waxes, etc. along with I. To obtain the water-soluble potassium copper chlorophyllin (II), which amounts to > 90% of chlorophyll production, this extract is saponified with 15-20% KOH in methanol. After phase separation, the hexane phase is further treated to obtain carotene, xanthophyll, and phytol, whilst the acetone-methanol-water phase, which contains potassium magnesium chlorophyllin, is washed with 90-95% hexane-acetone and then converted into the insoluble acid-copper chlorophyllin by acidifying with HCl to pH 1.5 and treating with $CuSO_4$ soln. After purifying by washing with 90-95% hexane-acetone and then with water, this is dissolved in KOH soln. Spray drying yields II in the form of beads of > 90% purity. Oil-soluble chlorophyll (I) is prepared by washing the initial hexane-acetone extract of the meal ten times with methanol and several times with water, and then evaporating to the required concentration. The Mg in I may be replaced by Cu by treatment with $Cu(NO_3)_2$ in acetone, giving an oil-soluble product of higher tinctorial strength and better fastness to light.

Chlorophyll a: $R = CH_3$ Chlorophyll b: $R = CHO$ 

W. K. R.

Pteridine Studies. V—The Monosubstituted Pteridines. A. Albert, D. J. Brown, and H. C. S. Wood. *J.C.S.*, 3832-3839 (Nov. 1954).

Electron-diffraction Study of Transformations of Thin Titanium Dioxide Layers by Change in Temperature. P. Conjeaud. *Compt. rend. Acad. Sci. Paris*, 238, 2076-2078 (1954); *Chem. Abs.*, 48, 13320 (25 Nov. 1954).

Layers of TiO_2 , 500-1000 Å. thick were obtained by vacuum distillation on to a cold surface. Amorphous TiO_2 is formed which is stable below 550°C. Above this temperature anatase and mixtures of anatase and rutile are formed. Above 800°C. only rutile is stable. No formation of brookite was seen. C. O. C.

Metallic Pigments. G. W. Wendon. *J.S.D.C.*, 71, 125-130 (March 1955).

The manufacture of metallic flake pigments is outlined. Their salient features are described and related to the more common applications. The production and the properties of dyed metal powders are discussed and contrasted with those of undyed metallic pigments as well as of ordinary pigments. Further examples are given of the combining of metallic appearance with spectral colours. AUTHOR

Hexagonal Tungsten Bronzes of Potassium, Rubidium, and Caesium. A. Magóli. *Acta Chem. Scand.*, 7, 315-324 (1953); *Chem. Abs.*, 48, 13508 (25 Nov. 1954).

Reduction of acid mixtures of WO_3 with K, Rb, or Cs tungstate yields a dark blue powder or hexagonal flaky crystals of M_xWO_3 (x has theoretical upper limit of $\frac{1}{2}$). The three compounds are isomorphous. The colour and the electrical conductivity of the three bronzes are in fair agreement with those of Na and Li tungsten bronzes.

C. O. C.

PATENTS

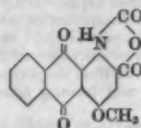
Reduction of Anthraquinone Derivatives. BASF. BP 720,738

Compounds containing ≤ 2 nuclearily combined CO groups, e.g. anthraquinones and dibenzanthrones, are reduced by treating them with nascent hydrogen in a melt of $AlCl_3$. Thus dibenzanthrone is added to a fused mixture of $AlCl_3$ and NaCl at 150-155°C. The complex $AlCl_3 \cdot 6NH_3$ is introduced followed by Zn dust. The melt is finally heated for 1 hr. at 170-175°C. and poured on to ice. After acidifying dibenzanthrone separates.

R. K. F.

Phthaloylisatoic Anhydrides. BASF. BP 719,193

A 1-aminoanthraquinone-2-carboxylic acid is treated with phosgene at elevated temperature to produce a phthaloylisatoic anhydride, useful as a dye intermediate. Thus the compound—



is prepared by passing phosgene into 1-amino-4-methoxyanthraquinone-2-carboxylic acid in nitrobenzene at 80°C. until no more is absorbed, and then cooling while passing in CO_2 .

R. K. F.

Monoazo Chrome Black for Wool. FBy. BP 721,719
The monoazo dye 3:5-dinitro-2-aminophenol \rightarrow 1:5-dihydroxynaphthalene-3:7-disulphonic acid dyes wool from an acid bath violet-black, becoming deep black on afterchroming.

E. S.

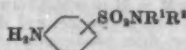
Monoazo Disperse Dyes. S. BP 721,632

Orange and red dyes for acetate, nylon and polyester fibres are made by coupling suitable diazo compounds with tertiary amines of the benzene series, in which the benzene nucleus contains at least 1 Hal atom, the N atom being attached to a cyanalkyl group as well as to an alkyl or hydroxyalkyl group. Thus the monoazo compound *p*-nitroaniline \rightarrow *m*-chloro-*N*-cyanoethyl-*N*-hydroxyethyl-aniline dyes acetate orange. The Hal atom is claimed to confer fastness to burnt gas fumes.

E. S.

Azoic Dyeings on Cellulose Acetate, Polyamide, and Polyurethan Fibres. FH. BP 720,520

Oranges and reds fast to cross-dyeing are produced on acetate, nylon and polyurethan fibre by applying in a weakly alkaline bath an anilinesulphonamide—



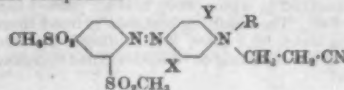
($R^1 = H$ or Alk; $R^2 = Alk$, Aralk, cycloalk or aryl; but R^1 must not = H if the sulphonamide group is *ortho* to the NH_2 , or if R^2 is Alk of $< 3C$; other substituents may be present in the benzene ring) together with an alkali metal compound of an arylamide of 3:2-hydroxynaphthoic acid containing an O Alk group in the arylamide group, alone or with other substituents. Diazotisation is then accomplished in cold aq. nitrous acid, and followed by development in a faintly alkaline bath. Thus cellulose acetate yarn (1000 g.) is treated for 1-1½ hr. at 75°C. in a bath obtained by pouring a hot soln. of the Na salt of 3:2-hydroxynaphtho-4'-methoxy-2'-methylanilide (18 g.) and 2:5-dimethoxyaniline-4-sulphonon-*n*-butylamide (20.6 g.) in diethanolamine (150 c.c.) and water (18 c.c.) into a mixture of hot water (30 l.), a condensation product (90 g.) obtained from oleyl chloride and albumen decomposition products, preferably linalbic acid (BP 413,016; *J.S.D.C.*, 50, 370

(1934)), 25% aq. ammonia (30 c.c.), and salt (600 g.). After diazotisation for 30 min. in a bath containing $NaNO_2$ (1 g./l.) and 20% B6. HCl (5 g./l.), the colour (claret) is developed by treatment at 60-80°C. in a bath containing oleylmethylaurine (1 g./l.), sodium acetate (4 g./l.), and 25% aq. ammonia (0.1 c.c./l.).

E. S.

Orange and Red Monoazo Dyes for Cellulose Acetate, etc. Eastman Kodak Co. USP 2,649,440

Monoazo compounds—

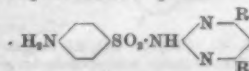


($R = \beta$ -hydroxyethyl, γ -hydroxypropyl, $\beta\gamma$ -dihydroxypropyl, β -sulphatoethyl, γ -sulphatopropyl or sulphated $\beta\gamma$ -dihydroxypropyl; $X = H$, CH_3 , Cl or Br ; $Y = H$, OCH_3 , OC_2H_5 , or OC_2H_4OH) are orange to rubine dyes for cellulose acetate, etc. Thus, 2:4-bismethylsulphonylaniline is diazotised in nitrosylsulphuric acid and coupled with *N*- β -hydroxyethyl-*N*- γ -cyanoethyl-aniline, to give an orange dye for cellulose acetate.

E. S.

Monoazo Compounds. Nepera, Chemical Co. Inc. USP 2,665,273

Brown azo dyes are made in substance or on the fibre by diazotising a sulphadiazine compound—

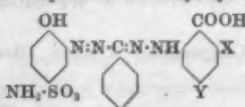


($R = H$ or CH_3) and coupling with a hydroxybenzoic acid. Thus the azo compound sulphadiazine \rightarrow *p*-aminosalicylic acid dyes cotton brown from an aqueous dispersion.

E. S.

Blue Metal(Copper)-complex Monoazo Dyes for Nylon, etc. DuP. USP 2,662,074

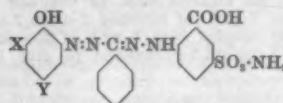
The copper complexes of formazyl dyes—



(X and $Y = H$ or Cl) give bright, deep blues on nylon and other hydrophobic fibres from a neutral bath. They are made by coupling diazotised 2-aminophenol-4-sulphonamide with the hydrazone made by condensing benzaldehyde with the hydrazine derived from anthranilic acid or its appropriate chloro derivatives. Or, as tautomeric rearrangement may interchange the positions of the azo and hydrazo links, diazotised anthranilic acid or its chloro derivatives may be coupled with hydrazone prepared from benzaldehyde and 2-hydroxyphenylhydrazine-5-sulphonamide. Thus diazotised 2-aminophenol-4-sulphonamide is coupled with a soln. of benzaldehyde *o*-carboxyphenylhydrazones in aq. NaOH, and the product converted to the metal complex by warming with ammoniacal copper acetate.

USP 2,662,075

Similar dyes, prepared in analogous manner, are made by converting compounds of formula—



(X and $Y = H$, Cl or NO_2) into copper complexes.

E. S.

Metal(Copper)-complex *oo'*-Dihydroxy-azo and -azomethine Dyes. BASF. BP 721,495

Copper complexes of *oo'*-dihydroxy-azo and -azomethine dyes are made by treating *o*-monoazo- or -azomethine compounds with elementary oxygen in presence of an oxygen carrier and cupric ions or metallic copper under weakly acid conditions. In BP 655,716 (*J.S.D.C.*, 67, 466 (1951)) and BP 660,447 (*ibid.*, 68, 90 (1952)) similar preparations are described using oxidising agents other than elementary oxygen. The starting materials

must contain a free α' -position, and at least one SO_3H group. The oxygen carriers employed include aldehydes, hydrazones, decahydronaphthalene and quinones like anthraquinone, hydroquinone, etc. By removing the copper from the complex with e.g. mineral acids or Na_2S , the free $\alpha\alpha'$ -dihydroxy compounds can be made. Thus, all parts being by weight, the monohydroxyazo dye (10) *p*-nitroaniline- \rightarrow R acid, sodium acetate (8), copper sulphate (5), and benzaldehydephenylhydrazone-4-sulphonic acid (7) are dissolved in water (800), and air passed through at 80°C . for 6 hr. The resulting complex is freed from copper by adding conc. HCl (100) and boiling. The chromium complex of the resulting $\alpha\alpha'$ -dihydroxyazo compound dyes wool blue. E. S.

Yellow Disazo Pigment. Interchemical Corpn.

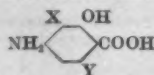
USP 2,657,204

The yellow disazo pigment 3:3'-dichlorobenzidine \rightarrow (acetacet-2:4-dimethoxyanilide), is similar in hue to the corresponding pigment derived from acetacet-*o*-toluidide, but of much superior light-fastness. E. S.

Chromable Mono- and Dis-azo Dyes for Wool. FH.

BP 719,631

4-Aminosalicylic acid and its derivatives—

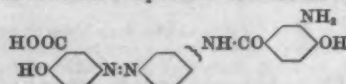


(X and Y = H, Cl, CH_3 or SO_3H) couple in alkaline solution with suitable diazo compounds to give monoazo dyes which may be metallised (particularly with Cr) in substance, on the fibre, or in the dyebath. Disazo dyes may be formed by diazotising these aminomonoazo dyes and coupling with suitable amines, phenols, naphthols, etc. Thus diazotised 4-chloro-2-aminophenol is coupled with 4-aminosalicylic acid dissolved in aq. NaOH . The product dyes wool from an acid bath dull yellow-brown, becoming red-brown when afterchromed or applied by the metachrome process. E. S.

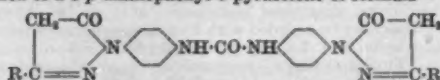
Metallisable (Copperable) Trisazo Direct Cotton Dyes. Gy.

BP 721,486

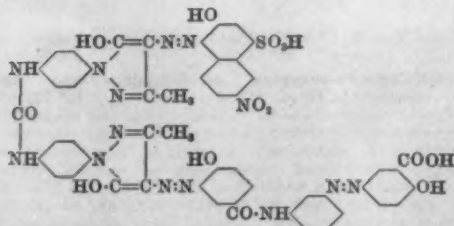
One mol. of a diazo compound of an aminoazo compound



and 1 mol. of the nitrated diazo compound of 1-amino-2-naphthol-4-sulphonic acid are coupled with 1 mol. of the urea of a 1-*p*-aminophenyl-5-pyrazolone of formula—



(R = CH_3 , C_2H_5 or phenyl) to give brown-orange direct cotton dyes suitable for after-coppering. Thus 3-amino-3'-carboxy-4'-hydroxyazobenzene is boiled in chlorobenzene with 4-hydroxy-3-nitrobenzoyl chloride, and the nitro group of the amide produced is reduced with Na_2S . The amino compound so produced is diazotised and coupled with an alkaline soln. of the azo compound prepared from 1 mol. of nitrated 1-diazo-2-naphthol-4-sulphonic acid coupled with 1 mol. of the urea of 1-*p*-aminophenyl-3-methyl-5-pyrazolone to give the trisazo dye—



E. S.

Naphthaquinoneimine Disperse Dyes. Celanese Corpn. of America.

BP 720,850

5-Amino-8-hydroxy-1:4-naphthaquinone-1-imine, prepared by treating 1:5-dinitronaphthalene with sulphur sesquioxide in H_2SO_4 , is condensed with an aniline, nuclear substituted with a hydroxyalkyl group, to give blue disperse dyes with good fastness to burnt gas fumes. Thus the naphthaquinoneimine referred to is heated for 2 hr. at 90°C . in 80% acetic acid containing sodium acetate with *m*-aminobenzyl alcohol. The product is separated by pouring into oil and water. R. K. F.

Antraquinonoid Substantive Dye Mixtures. S.

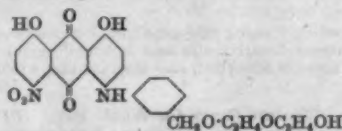
BP 719,177

Green substantive dye mixtures are prepared by treating 1-amino-4-aminoarylaminoanthraquinone-2-sulphonic acid, mixed with at least one related though different anthraquinone derivative and/or a yellow aminoazo dye, with the halide of a dicarboxylic acid. Thus 1-amino-4(*p*-aminophenylamino)-anthraquinone-2-sulphonic acid, the corresponding *o*- or *m*-methoxy derivative, the corresponding 6-sulphonic acid and the related 4(*p*-aminodiphenylamino)-6-sulphonic acid are dissolved in aq. NaOH , and fumaryl dichloride in CCl_4 added at 0°C . After distilling off the CCl_4 , the resulting dye is separated by making the solution strongly alkaline. R. K. F.

Hydroxy-Nitro-Polyalkoxyphenylamino-Anthraquinones—Disperse Dyes. ICI.

BP 721,885

α -Hydroxy- α' -nitroanthraquinones are condensed with anilines or toluidines containing in the *m*-position $-\text{CH}_2\text{O}(\text{RO})_n\text{Z}$, where R = alkylene of 2 or 3C, Z = H or Alk, and *n* = 1-3, to give blue disperse dyes fast to light and burnt gas fumes, and having affinity for polyester fibres. Thus the dye—

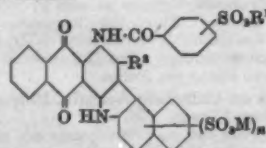


is made by heating 1:8-dihydroxy-4:5-dinitroanthraquinone with *m*-aminobenzyl- β -(β' -hydroxyethoxy)-ethyl ether at 120°C . for 3 hr. Separation is effected by cooling to 50°C . and diluting with 75% aq. ethanol. R. K. F.

Antraquinone Naphthocarbazole Sulphonic Acids—Acid Dyes. Allied Chemical & Dye Corpn.

BP 721,310

Dyes of formula—

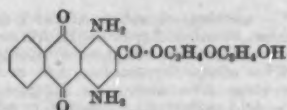


(R^1 = hydrocarbon radical containing 1-12C; R^2 = H, CH_3 , Hal or SO_3M ; M = H or a salt-forming radical; *n* = a positive integer) are brown neutral dyeing acid dyes. They are made by first condensing the corresponding 1-[(hydrocarbonsulphonyl)benzamido]-4-halogenanthraquinone with 2-naphthylamine and then cyclising the resulting compound to the carbazole. Thus 1-[*p*-(methylsulphonyl)benzamido]-2-methyl-4-chloroanthraquinone is heated at 170°C . for 10 hr. in *o*-dichlorobenzene with 2-naphthylamine in presence of K and Cu acetates and the product cyclised by stirring in H_2SO_4 at $30-40^\circ\text{C}$. until a sample is soluble in dil. aq. Na_2CO_3 . R. K. F.

Aminoanthraquinone Glycol Esters—Disperse Dyes. ICI.

BP 721,283

Disperse dyes of good fastness to light and burnt gas fumes on cellulose acetate, and with affinity for polyethylene terephthalate, are prepared by condensing an alkylene glycol of formula $\text{H}(\text{OCH}_2\text{CH}_2)_n\text{OH}$ where *n* > 3 with an anthraquinone-2-carboxylic acid containing in positions 1 and 4 either $-\text{NH}_2$, or $-\text{NH Alk}$, or groups, e.g. NO_2 or Hal, capable of being replaced by $-\text{NH}_2$ or $-\text{NH Alk}$. Thus the blue dye—



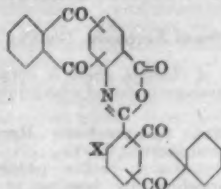
is made by heating 1-amino-4-nitroanthraquinone-2-carboxylic acid chloride with diethylene glycol at 100°C. for 1 hr. and reducing the product with Na_2S .

R. K. F.

Metoxazone Vat Dyes. BASF.

BP 720,795

Metoxazones of formula—



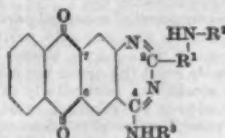
(X = Hal, NO_2 or NH_2) made according to FP 647,152 by the action of SOCl_2 in presence of FeCl_3 , on an *o*-aminoanthraquinone carboxylic acid are treated with ammonia or a primary amine to give vat dyes. In the reaction, the O of the metoxazone ring is replaced by NH or N-Alk. Thus the metoxazone derived from 1-aminoanthraquinone-2-carboxylic acid is heated under pressure at 130°C. for 5 hr. with 25% aq. NH_3 to produce a violet vat dye.

R. K. F.

Phthaloyl-quinazoline Vat Dyes. Ciba.

BP 719,670

Olive to brown vat dyes of formula—



(R^1 = an aromatic residue; R^2 = a vatable residue; R^3 = a cyclic residue containing at least one aromatic six-membered ring) and the carbazoles derived from them, are made by condensing the corresponding dihalogeno compound with the amines $\text{R}^2\text{-NH}_2$ and $\text{R}^3\text{-NH}_2$, either simultaneously or consecutively; and if desired cyclising the product to the carbazole. Thus 4-(anthraquinonyl-2'-amino)-2(2'-chlorophenyl)-6:7-phthaloyl-quinazoline is refluxed in nitrobenzene for 7 hr. with 1-amino-4-benzoylaminoanthraquinone, Na_2CO_3 and cupric acetate. The product may be cyclised by heating 1 hr. at 95–100°C. in a flux of SO_2 , AlCl_3 and NaCl .

R. K. F.

Liquid Indophenol Sulphur Dyes. Southern Dyestuff Corpn.

BP 720,440

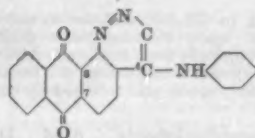
The precipitation and filtration stages following the sulphurisation of indophenols are avoided by having present in the sulphur melt the Na sulphinate of toluene, xylene, cymene, dimethylaniline or tetrahydronaphthalene. Yields are improved and the process simplified. Thus Hydron Blue is prepared by heating at 105°C. for 36 hr. a mixture of carbazole indophenol, diethylene glycol monoethyl ether, sodium *m*-xylenesulphonate, Na_2S and S. The resulting mass is used directly by diluting and adding Na_2S and NaHS to give the liquid sulphur dye.

R. K. F.

Phthaloylcinnoline Vat Dyes. BASF.

BP 719,906

4-Hydroxy-7:8-phthaloylcinnoline (made from 2-acetyl-1-aminoanthraquinone and HNO_3 as described in BP 709,614 (*J.S.D.C.*, 70, 369 (Aug. 1954)) and its substituted derivatives are treated with a cyclic aromatic primary amine in presence of agents capable of converting $-\text{COOH}$ to $-\text{CO-Hal}$ to give red brown, violet, olive and grey vat dyes of good levelling power. Thus the dye—



is made by first heating 4-hydroxy-7:8-phthaloylcinnoline with SOCl_2 in nitrobenzene at 80°C. until all has passed into solution. After distilling off excess SOCl_2 , aniline is added and the whole heated to 150°C. for 1 hr. The product is purified by dissolving in acetic acid, filtering and diluting the filtrate with water.

R. K. F.

Leuco Vat Dye Sulphuric Esters. DH.

BP 719,621

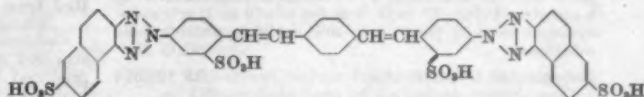
Leuco anthraquinone vat dyes which have hitherto been difficult to esterify are converted to the sulphuric esters by treating the parent vat dye in a mixture of *o*-picoline and diethylcyclohexylamine with chlorosulphonic acid in presence of finely divided Cu or brass. Thus, all parts being by weight, 1:4-dibenzylaminoanthraquinone (20) and Cu powder (15) are stirred for 4 hr. at 50°C. in a mixture of commercial *o*-picoline (150), diethylcyclohexylamine (55) and chlorosulphonic acid (20). The leuco ester is separated by pouring into aq. Na_2CO_3 , distilling off the bases, adding ethanol and NaOH at 35°C., and finally precipitating with NaCl .

R. K. F.

Triazole Dyes. Gy.

BP 718,962

A 1:4-bis(4'-aminostyryl)-benzene is tetrazotised and coupled with an amine which couples *o*- to the NH_2 group. The resulting diazo compound is oxidised to the corresponding bis-triazole, which is a greenish yellow dye for animal and vegetable fibres, dyeings on which fluoresce strongly yellow in ultraviolet. Thus, the dye—



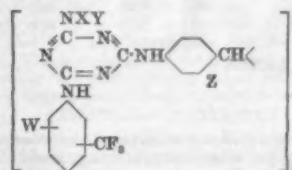
is made by tetrazotising 1:4-bis(4'-aminostyryl)-benzene 2':2'-disulphonic acid at 10–12°C., coupling with 2-amino-naphthalene-6-sulphonic acid and oxidising the product with CuSO_4 in presence of NH_3 at 92–97°C.

R. K. F.

Triazine Fluorescent Brightening Agents. General Aniline.

USP 2,666,052

Compounds of formula—



($\text{NXY} = \text{NH}_2$ or the residue of a primary or secondary amine, in which X and Y may together form a cyclic residue; W = H, alkoxy, aryloxy, Hal, cyano, trifluoroalkyl; Z = COOM or SO_3M (M = H or cation), e.g. that obtained by condensing 1 mol. of 4:4'-diamino-2'-stilbene-disulphonic acid with 2 mol. of cyanuric chloride, then with 2 mol. of 3-aminobenzotrifluoride and finally with 2 mol. of ammonia or a primary or secondary amine, are fluorescent brightening agents particularly suitable for incorporation in soap or other detergent.

C. O. C.

Magnesium Tetra-azaporphins. ICI.

BP 721,763

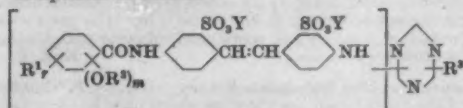
Magnesium tetra-aza-porphins, which are green, blue and violet pigments and which are convertible to the corresponding derivatives containing other metals, e.g. Ni, or no metal at all, are made by heating an alkyl-substituted fumaric nitrile with maleic dinitrile or one of its substituted derivatives, or with Δ^1 -tetrahydrophthalonitrile or phthalonitrile, along with a Mg alcoholate or the compound obtained by decomposing a Grignard reagent with an alcohol. Thus, *n*-amyl alcohol is added to a

solution of Mg in CH_3OH and the mixture distilled to b.p. 134–135°C. when dimethylmaleic dinitrile and dimethylfumario dinitrile are introduced. After refluxing for 2 hr. the amyl alcohol is distilled off and the crude pigment purified with 50% acetic acid followed by benzene.

R. K. F.

**Diaminostilbenedisulphonic Acid Derivatives—
Fluorescent Brightening Agents.** American
Cyanamid Co. BP 721,281

Compounds of formula—

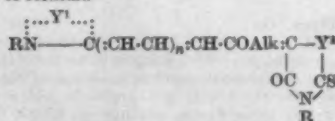


(R^1 and R^2 = Alk of 1–4C; R^3 = amino, alkylamino, arylamino; hydroxyalkylamino, alkoxyalkoxy, hydroxy, alkoxy or aryloxy; $r = < 3$; $m + 2r = < 4$; $\text{Y} = \text{H}$, NH , or alkali metal) are fluorescent brightening agents of good solubility, and affinity for the fibre and resistance to hypochlorite and not tending to discolour detergents.

C. O. C.

Di- and Tetramethinmerocyanine Dyes. Gevaert.
BP 721,203

Dyes of formula—

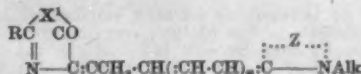


($\text{R} = \text{Alk}$, Ar or aralkyl; $\text{Y}^2 = \text{atoms to complete a 5- or 6-membered ring}$; $\text{Y}^2 = \text{O}$, S or Se ; $n = 0$ or 1) or rhodacyanines derived from them are valuable photographic sensitizers.

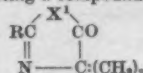
C. O. C.

Polymethin Dyes. Kodak. BP 720,556

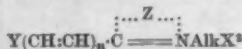
Dyes of formula—



($\text{R} = \text{Ar}$, styryl, alkylmercapto or arylmercapto; $\text{X}^1 = \text{O}$ or S ; $\text{Z} = \text{residue of a heterocyclic nucleus}$; $n = 0$ or 1) are obtained by reacting a compound of formula—



with one of formula—

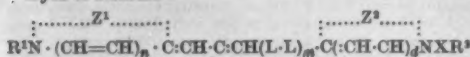


($\text{Y} = \text{a reactive group or atom capable of reacting with the } \text{CH}_2 \text{ group of the other compound, e.g. acetanilido or Hal}$; $\text{X}^2 = \text{anion}$). They have photographic sensitizing properties.

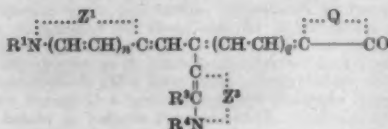
C. O. C.

Trinuclear Polymethin Dyes containing a Pyrrol, Indolyl, or Pyrrocolyl Nucleus. Eastman Kodak Co.
USP 2,666,761

Dyes of formula—



or



(R^1 and $\text{R}^3 = \text{unsubst. or subst. Alk of 1-5C}$; $\text{R}^2 = \text{H}$, Alk , or $\text{unsubst. or subst. Ar}$; $\text{R}^4 = \text{H}$, Alk or Ar ; R^5 and R^6 together = atoms to complete a pyridine nucleus; Q , Z^1 and $\text{Z}^2 = \text{atoms to complete a 5- or 6-membered heterocyclic ring}$; $\text{Z}^3 = \text{atoms to complete a pyrrole nucleus}$; $\text{X} = \text{acid radical}$; $\text{L} = \text{unsubst. or subst. methin}$; n and $d = 0$ or 1 ; m and $q = 0, 1$ or 2) are useful photographic sensitizers.

C. O. C.

Stabilising the Viscosity of Vat Dye Pastes. General
Aniline. USP 2,665,188

Stable aqueous vat dye pastes of low viscosity are produced by incorporating in them a little of a trialkylolamine triester of an aliphatic acid, e.g. triethanolamine triacrylate.

C. O. C.

Carbon Black from Aerosols. Godfrey L. Cabot.

USP 2,665,194

Carbon black of channel grade is obtained in greatly increased yield from gas enriched with liquid hydrocarbons.

C. O. C.

Tarnish-resistant Copper-base Bronze Powder. Malone Bronze Powder Works. USP 2,666,714

Copper or copper-zinc bronze powder is rendered resistant to tarnishing by heating to 600°C. in an atmosphere having no decarburising properties, e.g. in propane gas. The process is not applicable to aluminium bronzes which it renders more tarnishable but can be used to reduce the tendency to tarnish of silver, nickel and copper-nickel-zinc powders.

C. O. C.

Cuprous Oxide. Mountain Copper Co. USP 2,665,192

Cuprous oxide of high purity and very small particle size is obtained by treating an aqueous dispersion or solution of a copper compound with SO_2 and heating to convert the precipitated basic copper sulphite into cuprous oxide.

C. O. C.

Red Iron Oxide of High Colour Purity. Minnesota Mining & Manufacturing Co. USP 2,665,193

Red iron oxide composed of spherical particles of uniform size and of a hitherto unobtainable purity of colour is produced by heating an aqueous slurry of precipitated hydrous ferric oxide until the oxide has a constant colour value. Uniform conditions, particularly of the concentration of ferric chloride in the solution, must be maintained throughout the precipitation of the ferric oxide. Heating can be carried out as quickly as desired as the particle size and hue of the finished product are largely independent of the time and temperature of ageing.

C. O. C.

Complex Basic Ferric Carboxylate Pigments. Interchemical Corp. BP 721,749

Pigments having the transparency and colour range of organic pigments coupled with the light fastness of iron oxide pigments are produced by reacting ferric hydroxide with one of the following acids or its ammonium salt—a fatty acid of $> 3\text{C}$, chlorinated oleic acid, fatty acids of chlorinated cotton seed oil, 8:9-dichlorostearic acid or a naphthenic acid. The reaction is carried out in presence of a nonreactive compound containing both an organohydrophobic and a hydrophilic group, e.g. an alkyl, alcohol ester, ketone, glycol ether or amine. There should be 3 mol. of $\text{Fe}_2(\text{OH})_6$ to each mol. of the acid.

C. O. C.

Affinities of Vat Dyes in Relation to their Constitution (VIII p. 161).

Colour Development. III—Influence of Coupler on the Oxidation-Reduction Potential of the Developer (IX p. 162).

Bispyrazolone Colour Coupler (IX p. 162).

V—PAINTS; ENAMELS; INKS

Pigmentation of Flat Paints. W. H. Madson. *Paint & Varnish Production*, 44, (9), 21–24, 72 (1954); *Chem. Abs.*, 48, 14237 (25 Nov. 1954).

Uniformity of colour over surfaces of widely varying porosity is obtained with a pigment of rutile, selected CaCO_3 , and diatomaceous silica. Alkyds, linseed oils, modified oils, etc. can be used as the vehicle. A method of evaluating colour uniformity is given. Study of pigment-volume relationships indicated that a 65% polyvinyl chloride is best for general purpose flat paints. A "polyvinyl chloride ladder" technique is described, which assists

in selecting the best ratios of pigments. The effects of extenders on hiding power, brightness, and colour uniformity are considered, the importance of correct selection of extender being stressed, 0.5–1.0 lb. of diatomaceous silica per gal. (U.S.A.) of finished paint should be used. Data on the effects of Ti–Ca pigment blends are given. Causes of streaking in flat paints are discussed, and the special problems associated with use of odourless vehicles reviewed. C. O. C.

Black Cellulose Nitrate Chips. A. Weihe. *Farbe u. Lack*, 60, 398–402 (1954); *Chem. Abs.*, 48, 14240 (25 Nov. 1954).

Black gloss lacquers based on cellulose nitrate and carbon black offer many production problems because of the instability of the dispersion of ordinary blacks. Dispersing agents (thermoplastic resins, Cu oleate, Cu salts of acid phthalate esters, aromatic diamines, etc.) and chemical treatment of the blacks (oxidation to 8% O content, action of decomposing NH_4NO_3 or hydrazine salts, etc.) have helped to meet these problems. Efweko R is a German product consisting of cellulose nitrate chips containing lampblack of particle size 20 μ . It produces a highly glossy film. C. O. C.

Aluminium Alkoxides—New Group of Paint Additives. J. Rinse. *Paint Technol.*, 18, (204), 6–7 (1954).

Al alkoxides increase viscosity, remove water, decrease pigment settling, improve non-penetration, improve drying, decrease tendency to wrinkle, and improve resistance to water. Mixtures of fatty acids and Al alkoxides dry very rapidly in air. Up to 5% of Al isopropoxide improves the weathering properties of unbodied oil paints. C. O. C.

PATENT

Coating Solid Particles. D. C. Soul. *BP 721,467*
Solid particles of e.g. pigment are mixed either dry or as an aqueous slurry with a solution of an ammonium or volatile amine salt of a straight chain fatty acid of > 11C and dried at a temperature which decomposes the ammonium or amine salt. The process may be used e.g. to render pigments more readily dispersible. C. O. C.

Leaching of Aluminium–Asphalt Paints—Creep Test for Leaching (XIV p. 164).

VI—FIBRES; YARNS; FABRICS

Microscopical Study of the Effects of some Typical Chemical Environments on the Primary Wall of the Cotton Fibre. V. W. Tripp, A. T. Moore, and M. L. Rollins. *Text. Research J.*, 24, 956–970 (Nov. 1954).

The topography and the structure of the primary wall of the cotton lint fibre are described and illustrated by electron micrographs. The changes in this thin membrane brought about by alkali boil, hypochlorite and peroxide bleaching, mercerisation, acid hydrolysis, and dry heat are shown by means of microscopical examination of isolated pieces of the primary wall after treatment. The contribution of the primary wall to certain gross properties of the fibre and its behaviour during finishing treatments are discussed. A. B.

Morphological Relations in Cellulose Fibre Cells. F. Stern and H. P. Stout. *J. Textile Inst.*, 45, τ 896– τ 911 (Dec. 1954).

The average dimensions and orientations (as measured by the Hermans angle) of phloem fibre cells (from the stems) and of leaf fibre cells, each from 15 different varieties of plant, are measured, the principles of the X-ray method of determining orientation in native cellulose fibre cells being outlined. Leaf fibre cells show considerable variation in these properties, there being an interdependence such that the longer cells are of smaller width and steeper spiral than the shorter cells. Phloem fibre cells have different dimensions and orientation according to whether they are of primary or secondary origin, the former being longer, broader, and oriented more nearly parallel to the length. The relations connecting length, breadth, and orientation of the cells are interpreted in terms of a simple spiral model, and the mean number of

spiral turns per mm. are 10 for leaf, 4–5 for secondary phloem, and 2–3 for primary phloem. Coir is in a special category in having cells of entirely different orientation and dimensions. The constancy of the No. of spiral turns per millimetre of leaf fibre cells, irrespective of the external dimensions, indicates that, independently of the plant species, they have the same fundamental basis of structure. The secondary phloem fibres show remarkable uniformity of cell dimensions and orientation. The primaries show marked variation in width but not in length, suggesting that the width may be of more fundamental significance. J. W. B.

Properties of Cyanoethylated Cotton. AATCC Rhode Island Section. *Amer. Dyestuff Rep.*, 43, P 774–P 770 (22 Nov. 1954).

The effect of various commercial processes on cyanoethylated cotton, and the stability of cyano groups towards various treatments, including dyeing, printing, and finishing, are studied. The fibre (known as T 7) with an N content of 3.54% appears to have no marked advantages with regard to colour, depth, and fastness to compensate for the cost of the treatment. Sometimes the light fastness is reduced. Heat and bacterial resistance are improved. Better dyeing is effected by the Cu^+ ion process, but the fastness to washing is poor. It is possible that the rather unimpressive dyeing results may be due to the high N content, a lower value of 1.5% (at which, however, many other advantages are lost) having previously been recommended in this respect. The high N content, however, appears to improve disperse dyeing; though, apart from some improvement towards gas-fumes, it is doubtful whether satisfactory fastness can be obtained. Treatment of T 7 with alkali eliminates affinity for basic dyes, showing that the cyano groups are destroyed. Urea-formaldehyde resin treatment decreases the light fastness of direct dyes, but finishing treatments generally have little effect unless they are such that they hydrolyse the cyano groups. A method (essentially Kjeldahl) is appended for determining the N content. J. W. B.

Effect of Solvent–Solute Interaction on the Viscosity of Dilute Solutions of Cellulose Nitrate. T. E. Timell. *Svensk Papperstidning*, 57, 913–920 (31 Dec. 1954).

The influence of the solvent–solute interaction on the viscosity of dilute solutions of a high-mol.wt. nitrate of cotton cellulose was investigated with controlled rates of shear, using different solvents, and also varying the N substitution of the derivative with ethyl acetate as solvent. Similar results were obtained in the two cases, and it was found that the intrinsic viscosity was lower the poorer the solvent and the lower the N content. The shear rate dependence was primarily a function of the intrinsic viscosity, and was less the higher the degree of polymerisation. The importance of correcting for the degree of substitution of cellulose nitrates when estimating their molecular weight viscometrically is emphasised. S. V. S.

Fine Structure in Viscose Rayon Fibres. G. D. Joshi and J. M. Preston. *Text. Research J.*, 24, 971–979 (Nov. 1954).

Microscopical evidence is presented which shows that there are apparently four distinct zones in a viscose rayon fibre cross-section. There is a very thin outer zone which appears not to dye and has no double refraction. Within this there is a second layer, the so-called "skin", which is shown to have both radial and tangential orientations. The third layer absorbs more dye than the skin, but strips more easily. The innermost region takes up the most dye, but also strips most easily. A. B.

Influence of Inclusion on the Reactivity of Cross-linked and Non-crosslinked Cellulose Staple Fibres. H. Kriesig and E. Schrott. *Bull. Inst. Text. France*, (49), 47–60 (Oct. 1954).

It is possible to prepare rayon staples having different degrees of swelling, and thence, by replacing the swelling agent by certain organic solvents, different degrees of inclusion. It is shown that there exists a certain degree of inclusion below which the cellulose is not sufficiently reactive to be readily acetylated. This limiting degree of inclusion for acetylation depends on the molecular size of the included substance. A similar dependence of ease of

acetylation on degree of inclusion is found for staples cross-linked with hexamethylene diisocyanate. Although the degree of inclusion depends on the amount of cross-linking, the diurethane bridges themselves have little effect on acetylation. It is shown that the initial rapid rate of acetylation of cellulose containing included pyridine is not due to the preferential reaction of primary hydroxyl groups: the distribution of acetyl groups is statistical.

J. C. F.

Crystal Structure of Silk Fibroin. J. O. Warwicker. *Acta Cryst.*, 7, 565-573 (1954).

Evidence is given which indicates that silk fibroin has a structure having a twofold screw axis, and that it contains only one crystalline phase composed of glycine, alanine, and serine.

C. O. C.

Terminal Carboxyl Groups of Two Fractions of the Fibroin of *Bombyx mori* and of Whole Fibroin. J. T. B. Shaw and S. G. Smith. *J. Textile Inst.*, 45, T 939-T 940 (Dec. 1954).

Fibroin, and two fractions of fibroin obtained respectively by the action of chymotrypsin and of pancreatin mixed enzymes, are heated with anhydrous hydrazine, and the terminal α -carboxyl amino acid residues, being the only ones unaffected by the hydrazine, are separated by chromatography. The fraction from chymotryptic action gives large amounts of tyrosine and small amounts of phenylalanine, glycine, and alanine; that from mixed enzyme action gives glycine, alanine, tyrosine, and a trace of serine. These results confirm previous findings that proteolytic enzymes cause fractionation of fibroin, with precipitation of short side-chain amino acids and a little tyrosine; and that chymotrypsin causes molecular fission at the α -carboxyl group of the aromatic amino-acid residues of tyrosine and phenylalanine. Whole fibroin dissolves much less readily in hydrazine than the fractions, only 95% of the protein dissolving after 24 hr. at 110°C.; the C-terminal amino acids are found to be glycine, alanine, and smaller amounts of tyrosine, serine, valine, and proline. The fact that certain amino acids, including arginine, glutamic acid, aspartic acid, and tryptophan, have been stated to be wholly or partly decomposed by hydrazine treatment means that the values obtained do not necessarily represent all those present in the original fibroin. This consideration does not apply to the fractions which do not contain any of the amino acids attacked by hydrazine.

J. W. B.

Textile Properties of the Synthetic Fibres. T. H. Morton. *J. Appl. Chem.*, 4, 525-538 (Oct. 1954).

A survey is made of the qualities (physical, chemical, biological, economic) required in commercial textile fibres and of the extent to which these qualities are found in existing natural and synthetic textile fibres (tables of comparative physical data are given). Criteria useful in assessing a new fibre-forming polymer with respect to its textile utility are discussed.

A. E. S.

Detection and Isolation of the Skin of Polyamide Fibres. K. Schwertasek. *Faserforsch. und Textiltech.*, 5, 493-497 (Nov. 1954).

Silicon filaments (6-nylon manufactured in Czechoslovakia by a process in which polymerisation, spinning, and stretching form a continuous sequence) are treated on a microscope slide with sulphuric acid (26-27% by wt.), and the differential swelling of skin and core is observed. The concentration required to produce separation of the skin is rather critical, and differs according to the treatment that the fibre has received (fibres from dyed and set textiles give the best separation); it is best attained by observing the process under the microscope while making controlled additions of water or conc. H_2SO_4 . The skin may break away from the fibre and form a tight bowstring across a curved length of the smooth swollen core. When the process is aided by application of pressure to the cover glass, the whole of the core may be dissolved away, leaving the relatively intact skin. There are ten photomicrographs.

A. E. S.

Orientation in some Synthetic Linear Polymers. A. W. Kumm. *Faserforsch. und Textiltech.*, 5, 497-501 (Nov. 1954).

A low tension is applied to a fibre for ~ 20 sec., and then removed; after ~ 60 sec. a slightly higher tension is applied and removed; and so on, until breaking occurs. Values of the extension of the fibre in the loaded and un-

loaded states are plotted on one diagram against the load applied and removed, respectively, and the area bounded by these curves and the line representing the breaking load is expressed as a percentage E of the total area enclosed by the load-extension curve, the line representing the breaking load, and the load axis. The quantity E is the 'elastic component' of the work done in extending the fibre, and it is considered to be a measure of the average degree of orientation of the chain molecules in the fibre. Graphs for wool, silk, viscose rayon, Perlon, Orlon, and Dacron are given. The behaviour of polyacrylonitrile fibre and Perlon is studied in detail— E rises when the fibre is drawn, and, for a given degree of draft, the rise is greatest at low temp.; heat-setting causes a rise in E , whereas relaxation at high temp. results in a fall. The results are correlated with X-ray diffraction pictures of the fibres.

A. E. S.

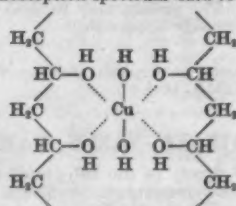
Fibres from Polyvinyl Alcohol. I. Sakurada. *Kolloid-Z.*, 139, 155-163 (Dec. 1954).

A description is given of the Japanese Vinyon fibres made from water-sol. polyvinyl alcohol, which are non-shrinkable and of considerable strength. The fibres are obtained from the sol. filaments by a hardening after-treatment, which consists of a combination of heating and acetylation. The description includes the preparation of polyvinyl alcohol, of the spinning solution and subsequent manufacture, of the stabilisation by after-treatment, and of hardening by formaldehyde. General properties are described with data, such as water-absorption, stability towards water and heat, physical properties, dyeing behaviour, and stability towards alkalis (ammonia), organic solvents, and oils. Future possible developments are discussed. 25 references and 6 diagrams.

H. H. H.

Adsorption of Copper by Polyvinyl Alcohol. S. Saito and H. Okuyama. *Kolloid-Z.*, 139, 150-155 (Dec. 1954).

Polyvinyl alcohol (average degree of polymerisation ~ 2100) combines with copper from weak alkaline soln. of $CuSO_4$ to give a green complex salt insol. in H_2O but sol. in NH_3 , whose composition is indicated from adsorption, viscosity, and absorption-spectrum data to be—



13 references and 5 diagrams.

H. H. H.

PATENTS

Regenerated Cellulose of Improved Colour by Removing Lead from the Coagulating Bath. Celanese Corp. of America. USP 2,666,684

Addition of an alkaline earth metal compound to the coagulating bath precipitates any lead present and so results in the regenerated cellulose obtained by using such a bath being of better colour.

C. O. C.

Coloured Foils or Fibres of Polyacrylonitrile. F. H. BP 720,632

A coupling component and a triazene compound of a diazo component are incorporated to the solution of polyacrylonitrile. After extrusion the dye is developed by the action of light and/or acid and/or heat.

C. O. C.

Aminated Acrylonitrile Copolymers having Good Affinity for Acid Dyes. American Cyanamid Co.

BP 721,688

Aminated copolymers of acrylonitrile, an ethylenically unsaturated aliphatic epoxy compound and one or more modifying comonomers are produced by copolymerising the monomers and then treating with ammonia or an amine. They are readily dyed with acid dyes.

C. O. C.

Whiteness Retention by Satin and Crêpe Lingerie Fabrics during Normal Wear and Cleaning (VII p. 161).

Imparting Affinity for Acid and Direct Dyes to Acrylonitrile Copolymers (X p. 163).

Kinetics of Axial Swelling of Viscose Rayon (XIV p. 164).

VII—DESIZING; SCOURING; CARBONISING; BLEACHING

Studies on Foams. VIII—Effects of Alcohols on the Foaminess of Soap Solutions. M. Nakagaki and K. Shinoda. *Bull. Chem. Soc. Japan*, **27**, 367-371 (Aug. 1954).

The foaminess of soap solutions increases with increasing concentration up to the critical micelle concentration and then remains almost constant. In a homologous series of potassium soaps, greatest foaming was observed at C_{12} - C_{13} . With mixed soap solutions, foaming decreased when the proportion of lower soap was greatest. On the addition of alcohols to soap solutions, three effects were noted—(a) defoaming by solvent action by C_2 - C_4 alcohols with K caprate at concn. $>$ soap; (b) foam promotion by C_6 - C_{10} alcohols with K caprate and C_8 - C_{10} alcohols with K caprate at concn. 0.001-1.0 of soap; and (c) defoaming by preferential adsorption by undecyl alcohol with K caprate and C_8 - C_{10} alcohols with K caprate at concn. $<$ 0.001 of soap. W. K. R.

Whiteness Retention by Satin and Crêpe Lingerie Fabrics during Normal Wear and Cleaning. C. H. Lindsey. *Amer. Dyestuff Rep.*, **43**, P 760-P 763, P 770 (8 Nov. 1954).

Rayon, silk, and nylon underwear fabrics, each including both satin and crêpe weaves, are submitted to wearing and laundering tests and are classified in order of desirability, the criteria including dimensional stability and whiteness retention, and the latter being assessed photoelectrically. The order best \rightarrow poorest is considered to be—nylon crêpe, nylon satin, rayon satin, rayon crêpe, silk satin, and silk crêpe. A comparative study is made of similar fabrics which are soiled artificially, and very similar results are obtained. The satins release soil to a greater degree than the crêpes. J. W. B.

Soiling of Fabrics in Contact with the Skin. AATCC Washington Section. *Amer. Dyestuff Rep.*, **43**, P 751-P 759 (8 Nov. 1954).

A technique for soiling fabrics through contact with the skin entails the wearing of a specially designed collar inside the ordinary shirt collar. Soiling is measured by means of the changes in colour and reflectance, and laundering is carried out using both a laboratory-prepared detergent (to avoid optical bleaching agents) and dry cleaning. Distribution of soiling is measured by scanning the collar with a pinpoint of light and recording the reflectance automatically. The Kubelka-Munk equation is used to calculate a figure for soil content and is found to be valuable in interpreting test results. A comparison of plain and starched cotton broadcloth, cotton oxford, and Dacron, and a preliminary comparison of nylon, rayon, and cotton broadcloth, are made. Cotton appears to soil less readily than nylon and Dacron, viscose rayon and acetate rayon being intermediate. The reflectance decreases rapidly at first and then more slowly, but the accumulation of soil is linear throughout the period of wear. Yellowing is of great importance with all fibres, and at least one popular detergent is found to contain an optical brightening agent effective in masking yellowing on cotton. It is found that the major fraction of soil is removed in the first minute of laundering, removal after 4 min. being negligible. An initial high soil content appears to result in a significantly higher residue than usual in the laundered sample. J. W. B.

Dry Cleaning of Cellulose Triacetate. E. J. Davies and W. K. Rhodes. *J.S.D.C.*, **71**, 143 (March 1955).

Perchloroethylene and white spirit are recommended for dry-cleaning cellulose triacetate textiles dyed with disperse dyes. Trichloroethylene may lead to bleeding of the dye and creasing and shrinkage of the fabric, probably owing to swelling of the fibre by the solvent. C. J. W. H.

PATENTS

Desizing Glass Cloth. Glass Fibers. *USP* 2,666,720 Glass cloth containing an oil-starch size is desized by treating first with a 5-20% aqueous solution of urea at a moderate temperature to remove the starch and then with either (a) caustic alkali, (b) an oxidising agent, (c) batch operation, or (d) in an autoclave with air or oxygen under pressure, to remove the oil. This treatment results in a

scrupulously clean glass cloth particularly suitable for combining with resinous compositions. C. O. C.

Microscopical Study of the Effects of some Typical Chemical Environments on the Primary Wall of the Cotton Fibre (VI p. 159).

Comparison of Loose Cotton and Sliver in Bleaching and Dyeing (VIII below).

VIII—DYEING

Comparison of Loose Cotton and Sliver in Bleaching and Dyeing. H. Kittel. *Z. gesamte Textilind.*, **56**, 1302-1311 (5 Nov. 1954).

Cotton bleached and dyed in sliver form is cleaner than when processed in the loose state. No seeds or other impurities are present, and the spinning qualities of the fibre are maintained. Sliver can be dyed with azoic, vat, sulphur, or direct dyes. B. K.

Affinities of Vat Dyes in Relation to their Constitution. R. H. Peters and H. H. Sumner. *J.S.D.C.*, **71**, 130-138 (March 1955).

The affinities of a series of anthraquinone and carbocyclic vat dyes have been determined. The attraction for the fibre appears to arise from two main causes—polar or hydrogen bonding and non-polar or van der Waals forces. The results are analysed in terms of these. It is found that, in a series of related compounds, a relationship exists between the affinity and the van der Waals forces as determined by the extinction coefficients of the dyes. AUTHORS

Influence of Temperature on Vat Dye Application.

M. R. Fox. *Amer. Dyestuff Rep.*, **43**, P 785-P 787, P 796 (22 Nov. 1954).

A brief survey is made of vat dye application, with particular reference to newer practices in which high temperatures are employed. J. W. B.

Dyeing of Cyanoethylated Cotton. H. A. Schuyten and J. W. Weaver. *Text. Research J.*, **24**, 1005 (Nov. 1954).

The dyeing of cyanoethylated cotton is discussed. It has been found that the methods employed in the dyeing of polyacrylonitrile fibres can be applied, including the use of basic dyes and acid dyes by the euprous-ion method. A. B.

Levelling Unevenly Dyed Material. AATCC

Philadelphia Section. *Amer. Dyestuff Rep.*, **43**, P 839-P 875 (6 Dec. 1954).

Dyed and undyed skeins of yarn are treated together in baths at different pH values, for different times, and in presence of different auxiliaries, to determine the amount of dye transfer from the dyed to the undyed material. Several pages of tabulated data are presented for nylon, dyneel, wool, Acrilan, Orlon, cotton, and Dacron. Certain auxiliaries consistently improve levelling, probably owing to their capability of forming addition products with the dye molecules. It is, however, emphasised that the best auxiliary must be chosen for each dye class and fibre. J. W. B.

Spun-coloured Viscose Rayon. L. L. Walmsley. *Amer. Dyestuff Rep.*, **43**, P 790-P 792 (22 Nov. 1954).

A brief review is presented of the cost and the properties of some spun-coloured viscose rayon, in which British practice and experience are studied with a view to assessing the value of this method in America. J. W. B.

Improved Washing Fastness of Dyed Viscose Rayon-Acetate Rayon Blends. V. S. Salvin, A. B. Couciatori, R. A. Walker, G. C. Ward, and F. Fortson. *Amer. Dyestuff Rep.*, **43**, P 764-P 770 (8 Nov. 1954).

Principles underlying the fixation of acetate-rayon dyes by formaldehyde resins and copper fixatives are established; the dyes containing amine or aliphatic -OH groups interact with the hydroxymethyl groups of the resin, and Cu fixatives react by complex formation between Cu ions and chelatable groups when present in the anthraquinone structure. A process which can be used on viscose-acetate rayon or viscose-acetate rayon-nylon blends to give fastness to repeated machine washing at 120-130°F. is described, and, using various proprietary resins and a copper fixative, e.g. Cuprofix 47, it can be applied by the usual equipment. Data are presented on such factors as optimum concentration of reagents and catalysts, and on

the author finds that with viscose rayon fabrics leading beyond an optimum resin content, e.g. 10% for urea-formaldehyde, is harmful. Similar effects are shown for melamine-formaldehyde, ketone-formaldehyde, glyoxal, and formaldehyde treatments. The use of a syrup-type resin as stiffener is not detrimental provided that the optimum resin content is not exceeded, and in small amounts these resins form a protective coating against abrasive action. The factors involved in a good resin treatment are discussed and the amine-type catalysts, AC and G8, are recommended. The importance of washing-off all resin-treated fabrics is emphasised. W. G. C.

Silicone Resins in Textiles. F. Fortess. *Ind. Eng. Chem.*, 46, 2325-2331 (Nov. 1954).

Optimum conditions for the application of silicone resins to textiles, particularly acetate rayon, are described, and sources of difficulties are outlined. The necessity is stressed for complete removal of wetting agents as well as oils and sizes before treatment. Silicones based on liquid methylsiloxane polymers containing reactive groups for subsequent cross-linking are the most suitable. Fabrics are padded in an aqueous emulsion of the silicone, and then dried and cured, when the film is insolubilised by the formation of oxidative cross-links. With acetate rayon and Daeron, there is no evidence of reaction between the silicone and the fibre. Treatment provides durable water repellency, improved tear strength and abrasion resistance, and better sewing properties and recovery from wrinkling. W. K. R.

Adhesion to Vinyl Resins of Glass Fibres designed for the Production of Coated Articles. J. Pinte, P. Rochas, and M. Coupez. *Bull. Inst. Text. France*, (49), 7-32 (Oct. 1954).

Possible ways of improving the adhesion between glass and resin in resin-coated glass fabrics have been studied. Adhesion is improved to some extent by mechanical abrasion of, or superficial chemical attack on, the glass, but the mechanical properties are impaired. For loose-textured fabrics, treatment of the glass with a non-ionic wetting agent improves the adhesion by assisting penetration by the coating paste. The most marked increases in adhesion (400-500%) are obtained by treating the glass with a polyfunctional reagent (hexamethylene- or toluylene-bisethylenes) and incorporating reagents of the same type in the coating paste. J. C. F.

Flammability and the Law in the U.S.A. H. E. Hager. *Amer. Dyestuff Rep.*, 43, P 788-P 789 (22 Nov. 1954).

PATENTS

Protecting Cellulose against Heat Ageing. United States Rubber Co. *USP* 2,665,230

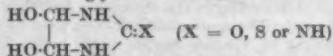
Cellulose, especially regenerated cellulose, is protected against degradation by heat ageing by impregnating it with an organic amine of b.p. < 75°C. and m.p. > 150°C., e.g. *o*- or *m*-phenylenediamine.

USP 2,665,733-4

Impregnating cellulose tyre cords with 0.1-4.0% of melamine or 0.1-10.0% of diacydiamide gives substantial improvement in resistance to heat ageing. C. O. C.

Crease-resistant and Water-repellent Finish for Cellulosic Textiles. BASF. *BP* 720,386-7

Treatment with solutions of methylol compounds of monourea in presence of an acid catalyst is used. The methylol compounds can be applied as such or may be formed by reaction between the monourea and formaldehyde, under neutral or alkaline conditions, in the fibres. The monourea of glyoxal of formula—



are particularly suitable; they form methylol compounds by combining with 1 or 2 mol. of HCHO. C. O. C.

Delayed-action Catalysts for Use in Formaldehyde Resin Finishes. Joseph Baneroft & Sons Co.

BP 720,016

Delayed catalysis is obtained by adding H₂O₂ to the resin liquor so that during hot mechanical treatment and/or baking the HCHO present is oxidised to formic acid which imparts sufficient acidity for correct curing of the resin. If necessary, HCHO may be also added to the liquor to serve as a reservoir in addition to that present in the resin mix. C. O. C.

Shrinking Textile Fabrics. L. Squarenina. *BP* 720,146
Fabrics, particularly wool fabrics, are treated in a closed chamber with an artificial mist and are then kept in a second closed chamber at constant temperature and R.H.

C. O. C.

Imparting Affinity for Acid and Direct Dyes to Acrylonitrile Copolymers. CFM. *BP* 720,024

Copolymers of acrylonitrile and vinyl esters of aliphatic α -halogeno carboxylic acids are given affinity for acid and direct dyes by treating them with hot aqueous solutions of aliphatic bases, e.g. 1-5% aqueous *NN*-diethylethylenediamine at 90-95°C. for 2 hr. C. O. C.

Application of Metal Films by Vacuum Evaporation. National Research Corp. *USP* 2,665,223-9

Application of metal films, particularly aluminium, to flexible surfaces by vacuum evaporation is described.

C. O. C.

Carrotting. E. Elöd. *BP* 721,716

The fur is removed from the skins, formed into bats and then impregnated with a carrotting agent. This enables carrotting liquors of less than conventional strength to be used and produces hat bodies freer from faults than those made from fur carrotted upon the skins or in a loose state.

C. O. C.

Velour Finishing of Embossed Felt Hat Bodies. I. Kaufman. *BP* 721,722

The embossed hat body is treated with a fine sharkskin surface (400-500 teeth per sq. in.) moving at low speed, e.g. 1250-1750 ft. per min. in a direction opposite to the nap of the sharkskin so as to open up the surface of the felt. It is then similarly treated with a coarser sharkskin surface (300-350 teeth per sq. in.) moving faster than the other one e.g. at 3750-5000 ft. per min. The felt is finally polished by treating with a fine sharkskin surface moving in the direction of the nap of the sharkskin. This very quickly imparts a highly lustrous, uniform and smooth finish. C. O. C.

Microscopical Study of the Effects of some Typical Chemical Environments on the Primary Wall of the Cotton Fibre (VI p. 159).

Pigment-binding Power of Textiles (VIII p. 162).

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Cellulose Studies. XIX—Distribution of Methoxyl Groups in Partly Methylated Celluloses. L. Rebenfeld and E. Pasou. *Text. Research J.*, 24, 941-955 (Nov. 1954).

The distribution of methoxyl groups in methylcellulose prepared by homogeneous, heterogeneous, and commercial methylation is investigated. It is shown that different products are obtained, depending upon the method used in preparation. The different mechanisms assigned to the cellulose methylation reaction, depending upon whether it is heterogeneous or homogeneous, are fully discussed.

PATENTS

Sheets of Paper or Felted Fibrous Material. S. M. Hjelto. *BP* 720,006

Fibrous pulp is fed to a sieve-like travelling conveyor belt so as to form a layer on it, the forming layer subjected to strong suction, filler being added to the pulp immediately before or during the suction application, and then weaker suction applied to the layer. S. V. S.

Sizing Paper. Shell Development Co. *USP* 2,665,206

A mixture of salts of naphthenic acids of acid number < 200 with conventional sizes or preferably with the predominantly cyclic extracts obtained from mineral oils is an excellent size for paper which is required to be resistant to water.

USP 2,665,207

Naphthenates of acid number > 200, preferably 200-400, are used either alone or in conjunction with other sizing agents. C. O. C.

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Structural Units in Collagen Fibrils. A. C. T. North, P. M. Cowan, and J. T. Randall. *Nature*, 174, 1142-1143 (18 Dec. 1954).

X-Ray diffraction evidence suggests that dry collagen fibres contain small crystalline regions, perhaps 50 Å. long,

within which the polypeptide chains have a helical configuration; these regions are arranged to give a 640 Å. macroperiod with an apparent 200 Å. substructure.

J. W. B.

PATENTS

Tanning. Verband der Deutschen Lederindustrie.

BP 720,505

The skins are impregnated with a monomeric polymerisable substance, e.g. a vinyl compound, which is either simultaneously or subsequently polymerised within the skins. This yields a soft leather which is highly resistant to water.

C. O. C.

Coating Composition for making Patent Leather.

Indian Council of Scientific and Industrial Research.

USP 2,666,717

The resin obtained by copolymerising bhillawan shell liquid with vegetable oils and with rosin and turpentine by treating with nitric acid at 40–50°C. is dissolved with film scrap in butyl acetate and solvent naphtha to yield a coating composition suitable for making leather. It yields a film having good adhesion and which is unaffected by changes in atmospheric humidity and temperature.

C. O. C.

Rapid Testing and Numerical Evaluation of the Covering Power of Casein and Collodion Pigment Colours (XIV this page).

XIII—RUBBER; RESINS; PLASTICS

Adsorption of Copper by Polyvinyl Alcohol (VI p. 160).

Coloured Foils or Fibres of Polyacrylonitrile (VI p. 160).

Aminated Acrylonitrile Copolymers having Good Affinity for Acid Dyes (VI p. 160).

Application of Metal Films by Vacuum Evaporation (X p. 163).

XIV—ANALYSIS; TESTING; APPARATUS

Gravimetric Determination of Cholesterol in Wool

Wax. H. W. Knol. *Rec. Trav. chim.*, 73, 924–930 (Nov. 1954).

Chromatography of Dye Intermediates. I—Paper Chromatography of Naphthylaminesulphonic Acids.

J. Latinák. *Chem. Listy*, 48, 843–846 (1954); *Chem. Abs.*, 48, 14213 (25 Nov. 1954).

Naphthylaminesulphonic acids can be separated by their R_f values in the systems butanol-acetic acid-water (4:1:5) and butanol-pyridine-water (3:1:1), which are as follows—Koch acid 0.00, 0.00; Freund acid 0.02, 0.04; amino-ε acid 0.14, 0.08; 1-naphthylamine-2-sulphonic acid 0.57, 0.63; 1-naphthylamine-3-sulphonic acid 0.41, 0.47; naphthionic acid 0.29, 0.42; Laurent acid 0.26, 0.44; Cleves acid (6-sulphonic) 0.34, 0.48; Cleves acid (7-sulphonic) 0.46, 0.54; peri acid 0.69, 0.63; α-naphthylamine 0.95, 0.92; 2-amino-R acid 0.03, 0.08; C acid 0.04, 0.06; 2-naphthylamine-1:6-disulphonic acid 0.03, 0.05; 2-amino-G acid 0.03, 0.04; Tobias acid 0.55, 0.61; Dahl acid 0.34, 0.47; Bröner acid 0.32, 0.46; 2-amino-F acid 0.33, 0.47; Baden acid 0.48, 0.51; β-naphthylamine 0.94, 0.92.

C. O. C.

General Method for Determining Diazo Compounds, especially the Fast Salts (Stabilised Diazo Compounds from Amines containing no Solubilising Groups).

L. Nicholas, P. Lampel, and J. Planté. *Chim. anal.*, 36, 238–241 (1954); *Chem. Abs.*, 48, 13544 (25 Nov. 1954).

One gram. of sample is heated with 150 ml. of 7.4N-H₂SO₄ under an atmosphere of CO₂. The N of the diazo compound is liberated as gas and is measured in a conventional azotometer.

C. O. C.

Evaluation of the Hiding Power of Pigments.

J. Pick. *Chem. Průmysl*, 10, 352–356 (1953); *Chem. Abs.*, 48, 14239 (25 Nov. 1953).

Make a paste of d g. (= 1 cc.) of pigment and 1.2 g. blown castor oil, and a lacquer of 20 g. low-viscosity cellulose nitrate, 10 g. ethanol, 25 g. xylene, 30 g. glycol ethyl ether, 5 g. dibutyl phthalate, and 5 g. cyclohexanone resin

AW2 dissolved in 5 g. xylene. Mix ($d + 1.2$) g. of the paste and 12.0 g. of the lacquer, apply to a glass plate, allow to dry, strip, and determine the hiding power on a contrast scale. A double film is not the same as twice the thickness of a single film. Formulae are given to compute percentage by weight and volume of dry substance in paint, pigment in paint, and pigment in dry substance from known constants of the constituents and the density of the pigment tested. The final interpretation of the results is the number of grams of the pigment under test having the same hiding power as 1 g. of standard sample. C. O. C.

Rapid Testing and Numerical Evaluation of the Covering Power of Casein and Collodion Pigment Colours.

O. Zohlen. *Leder*, 5, 187–189 (1954); *Chem. Abs.*, 48, 14238 (25 Nov. 1954).

Make a stripe 0.5 cm. wide with black Indian ink on white paperboard (6 × 10 cm.). On this place a round flat-bottomed crystallising dish 8 cm. in diameter and add to it 50 ml. of water (for casein) or mixed solvent (for collodion colours). The liquid must be exactly 1 cm. deep; the area of the bottom of the dish must be exactly 50.25 sq. cm. Dilute 1 or 2 g., depending on hiding power of the colour under test, to 100 ml. with water or solvent, then add this suspension slowly (finally dropwise) from a graduated pipette to the liquid in the crystallising dish until the black stripe cannot be seen after stirring. The effective covering power is the reciprocal of the number of ml. of 1% colour suspension per 100 sq. cm. of dish surface required to hide the stripe. The dish may be placed either directly on the paper with the stripe or raised above it by an inverted crystallising dish. Reproducible results are got by either method, although they may be higher by either.

C. O. C.

Leafling of Aluminium-Asphalt Paints—Creep Test for Leafling.

R. H. Frederick and W. P. Wooley. *Paint & Varnish Production*, 44, (8), 28–33 (1954); *Chem. Abs.*, 48, 14237 (25 Nov. 1954).

Leafling was determined by the creep test in which 45 g. Al paste is put in a can (capacity 118 ml.) and the asphaltic vehicle stirred in slowly until the can is full. After standing overnight the sealed can is opened and the paint stirred and poured into a 100-ml. Berzelius beaker so that the whole surface from top downwards is wetted with the paint. The beaker is then inverted for 20 sec. and stood for 30 min. at 80°C. until all leafling up the sides of the beaker has ceased. The distance from the top of the liquid left in the beaker to the first break in the leafling film on the side of the beaker is measured in mm. and known as the *creep*. The effects of asphalt composition, viscosity, sp. gr., and solvents used are considered, and the requirements for best leafling properties deduced from them.

C. O. C.

Quantitative Spectrographic Analyses of Metallic Elements in Textile Materials.

L. C. Block and D. C. W. Kwok. *Text. Research J.*, 24, 990–999 (Nov. 1954).

Four methods are described for spectrographic quantitative analysis, namely the internal-standard method, the modified internal-standard method, the logarithmic-sector method, and the addition method. It is shown that quantitative analysis of textile materials for their metallic content can be accomplished very expeditiously.

A. B.

Kinetics of Axial Swelling of Viscose Rayon.

J. Joly. *Bull. Inst. Text. France*, (49), 33–46 (Oct. 1954).

An apparatus is described which permits continuous photographic recording of the changes in length of a viscose rayon filament. The apparatus has been used to study the axial swelling of viscose rayon, both before and after mechanical extension, in water and in caustic soda solutions. It is shown that in certain cases the swelling behaviour is complex. The significance of the results is discussed.

J. C. F.

Morphological Relations in Cellulose Fibre Cells (VI p. 159).

Properties of Cyanoethylated Cotton (VI p. 159).

Effect of Solvent-Solute Interaction on the Viscosity of Dilute Solutions of Cellulose Nitrate (VI p. 159).

Soiling of Fabrics in Contact with the Skin (VII p. 161).

Pigment-binding Power of Textiles (VIII p. 162).

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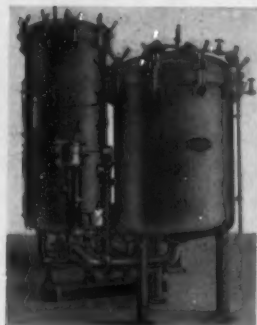
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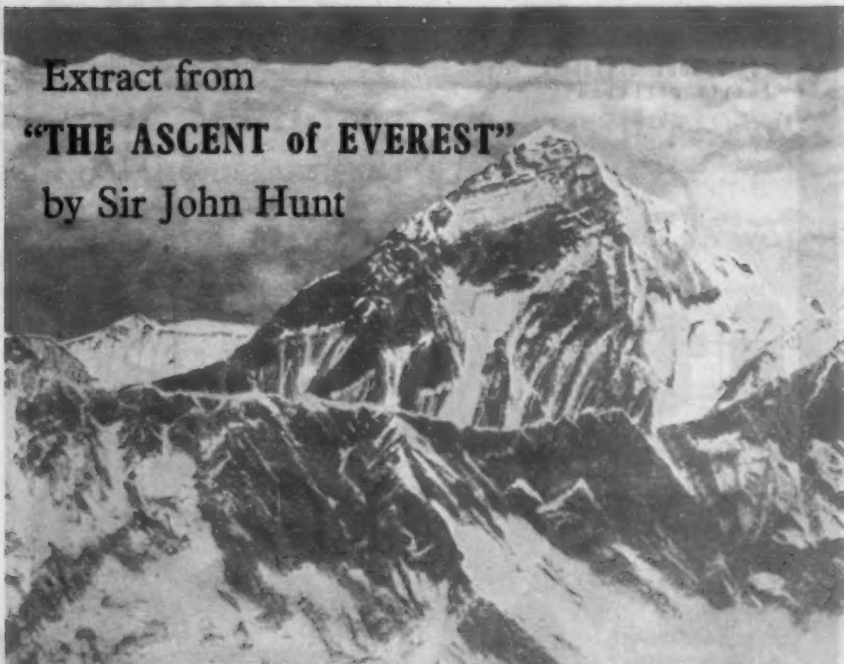
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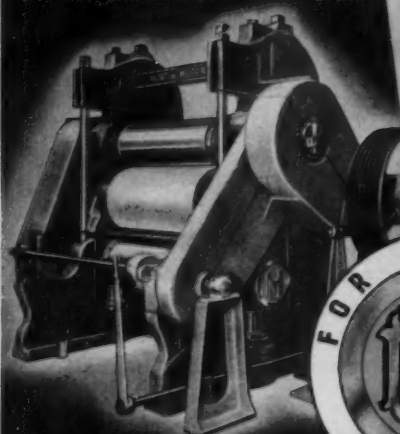
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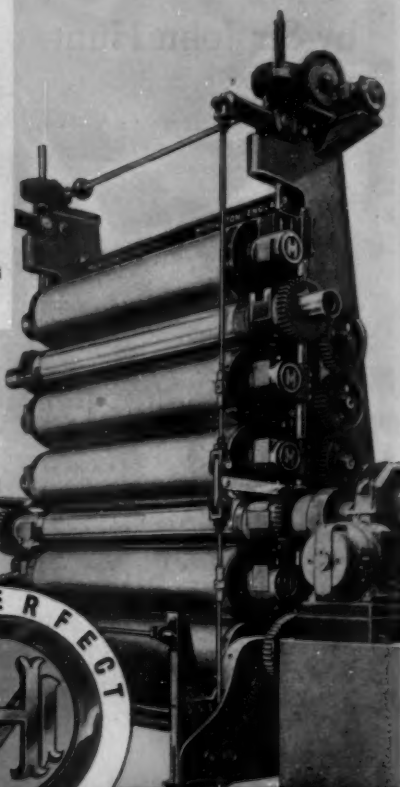
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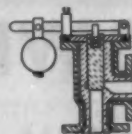
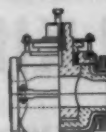
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